

3.4.3.2. Scanning electron microscope examination of the bending part of the three and four point bending samples

The tensile surfaces of the bending specimens were the most sensitive parts since they were under the highest tensile stress [3.4]. Hence, the crack will start to form at these points in the bending samples. The SEM micrographs taken at these points will help show whether the crack forms when the wire is bent. Under the same flexure level, the amount of crack formation is proportional to the brittleness of the wires.

The micrographs of the tensile sides of the surfaces of WF, WS, GF, and GS wires after 3 point bending tests are shown in Fig.3.23, 3.24, 3.25 & 3.26. Fig. 3.27, 3.28, 3.29 and 3.30 are the micrographs of the tensile sides of surfaces of WF, WS, GF and GS wires after 3&4 point bending tests. From these figures, we can see that WF wires are the most brittle since they show cracks on their tensile surfaces in both 3 and 4 bending tests.

The extent of the brittleness of the wires can be determined from the above micrographs.

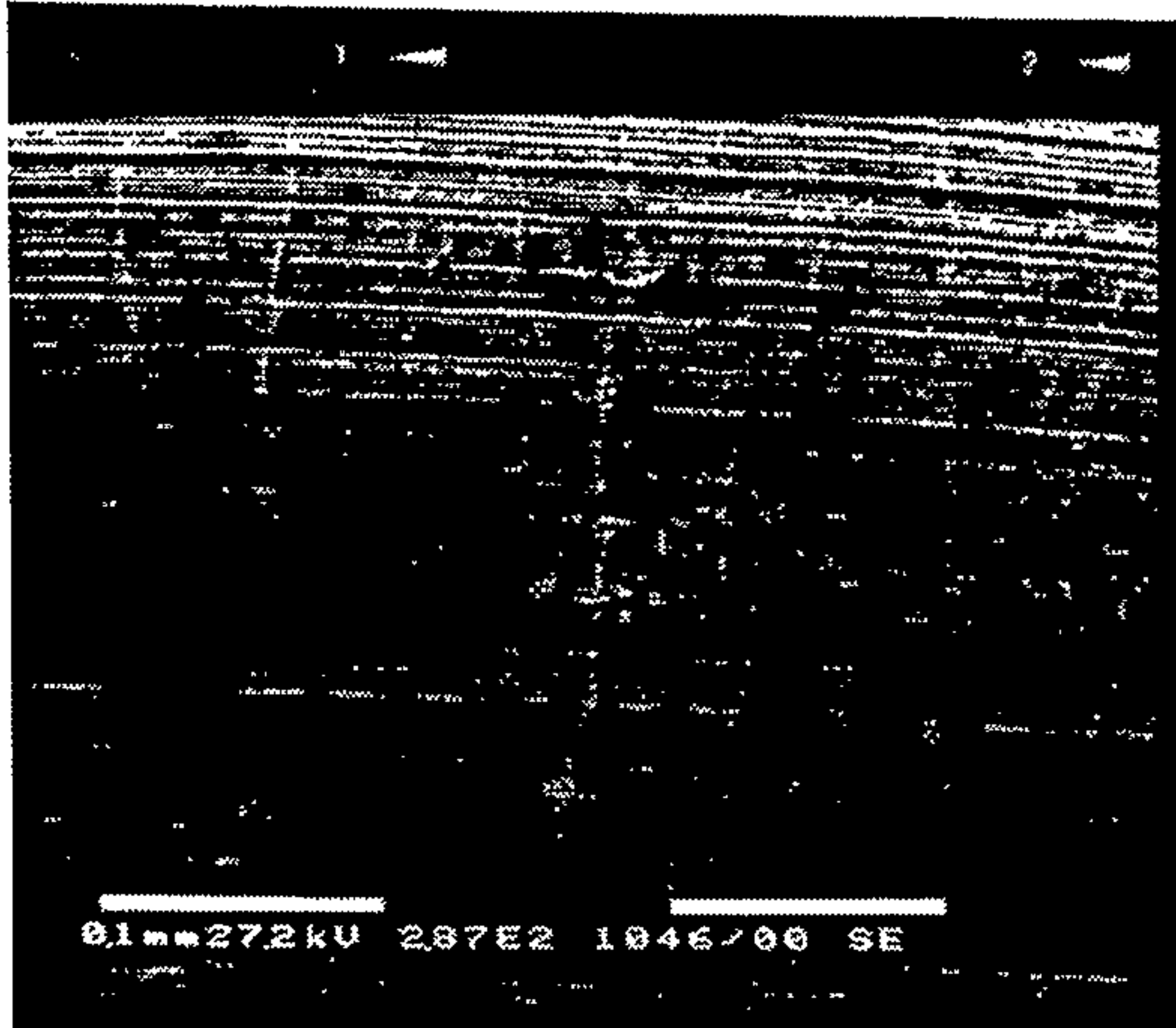


Fig. 3.23. Tensile surface of 3 point bending part of WF wire.

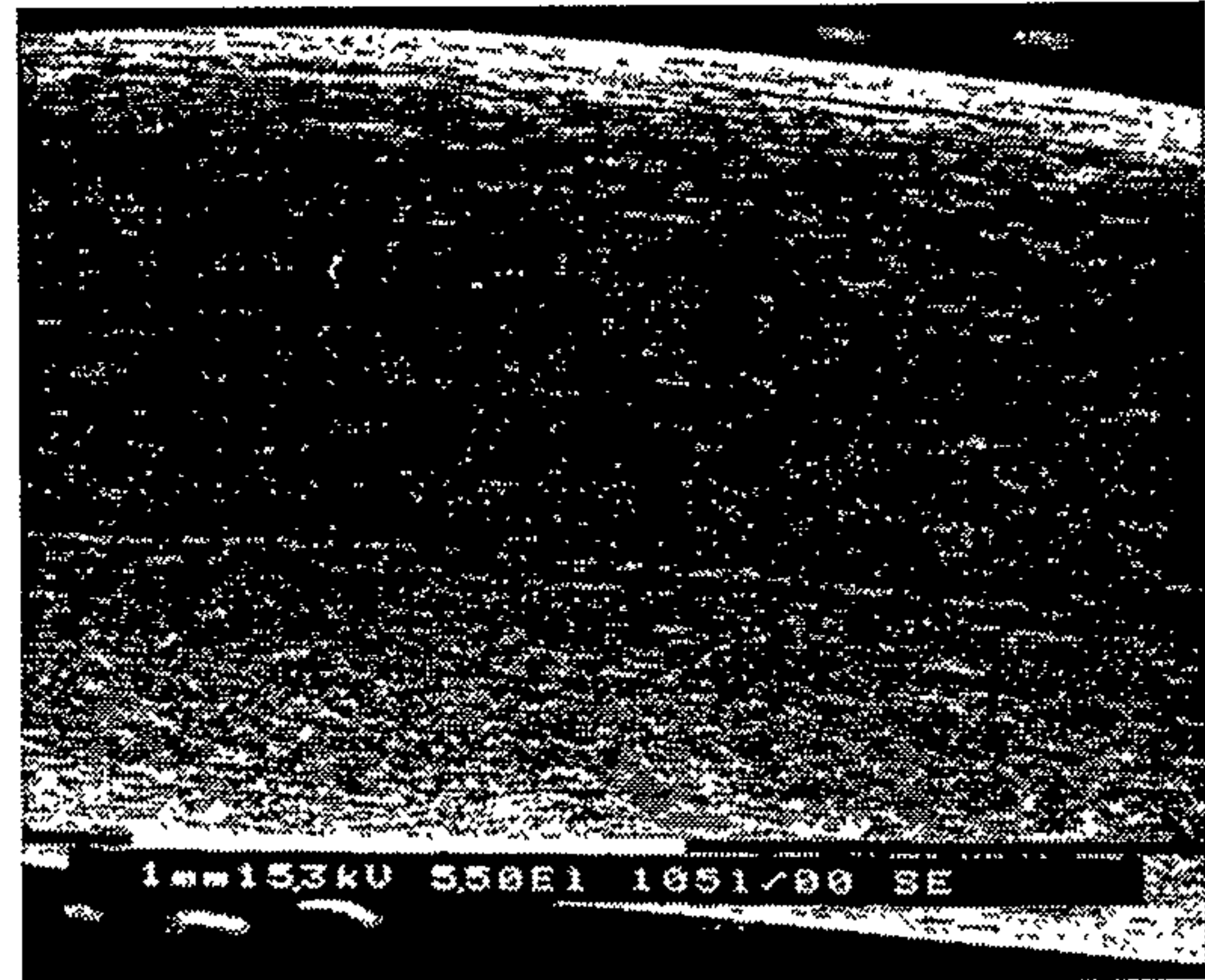


Fig. 3.24. Tensile surface of 3 point bending part of WS wire.

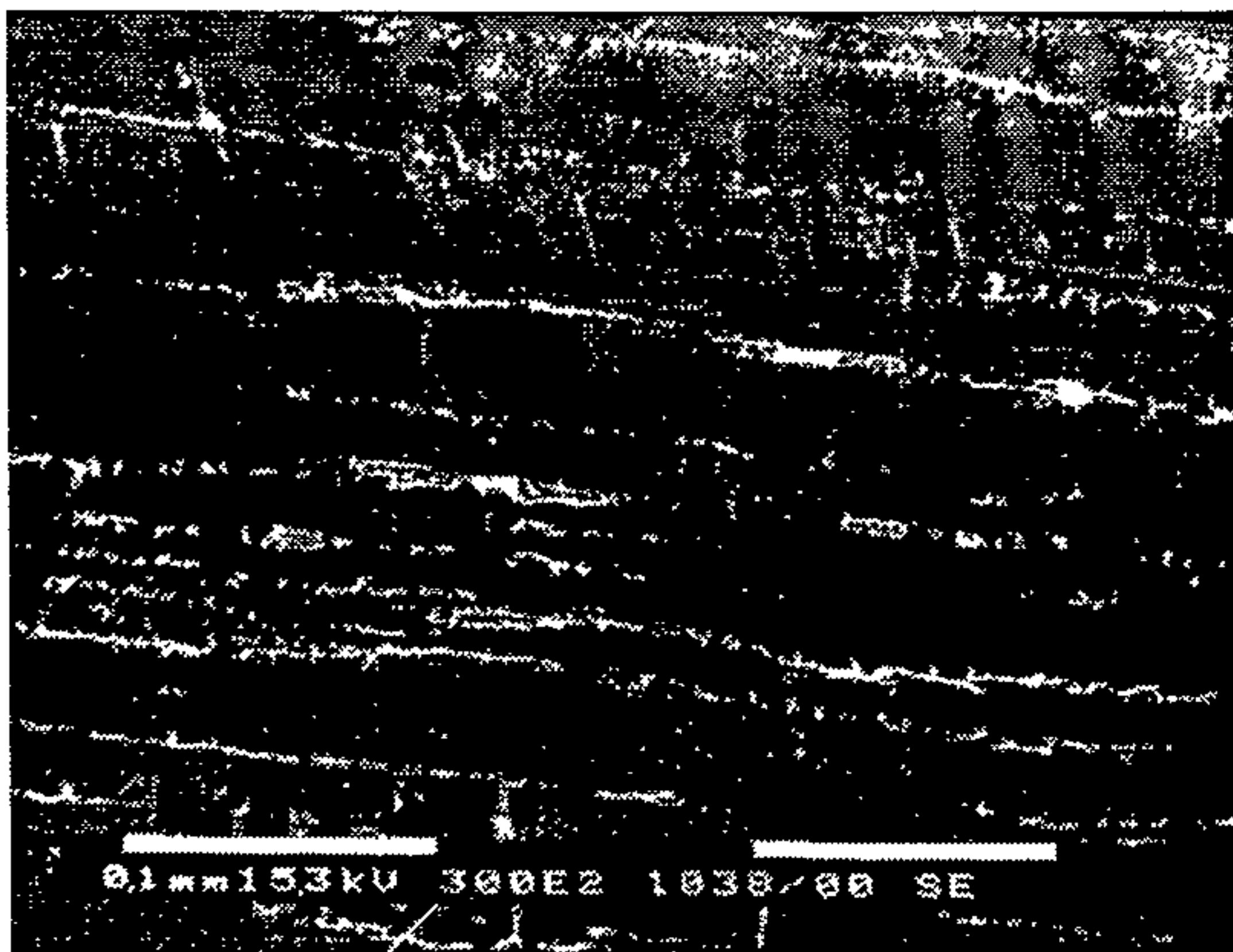


Fig. 3.25. Tensile surface of 3 point bending part of GF wire.

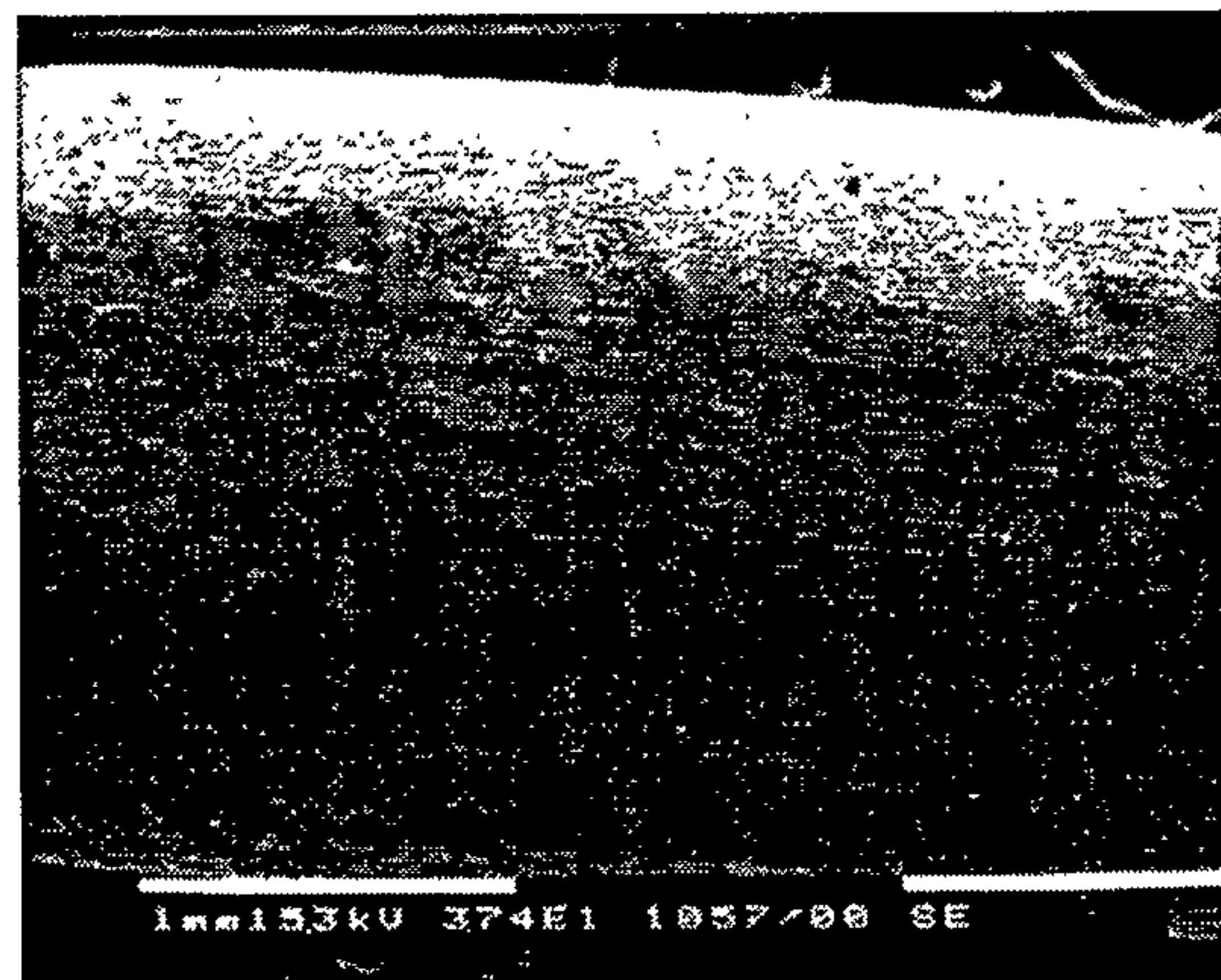


Fig. 3.26. Tensile surface of 3 point bending part of GS wire.

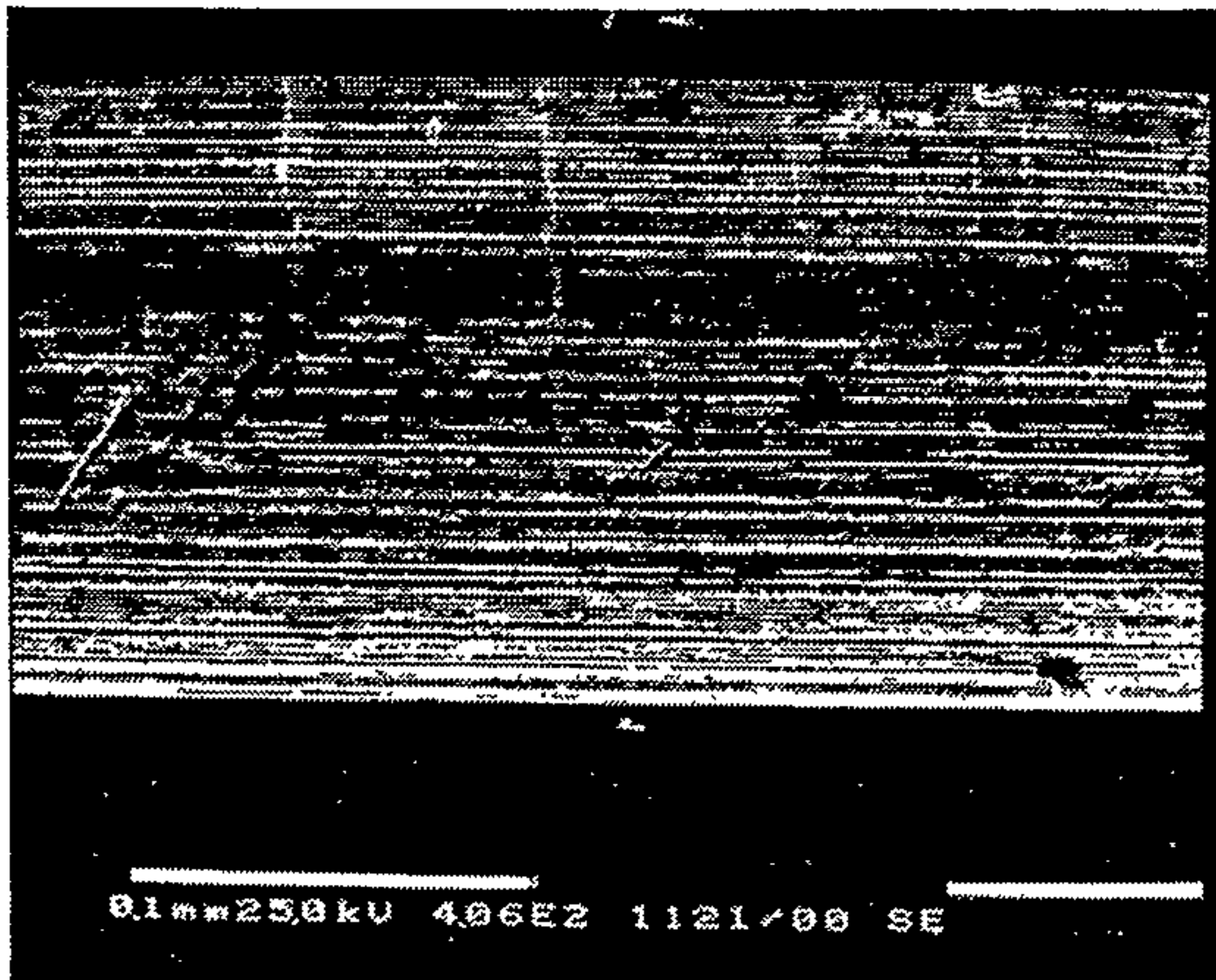


Fig.3.27. Tensile surface of 4 point bending part of WF wire.

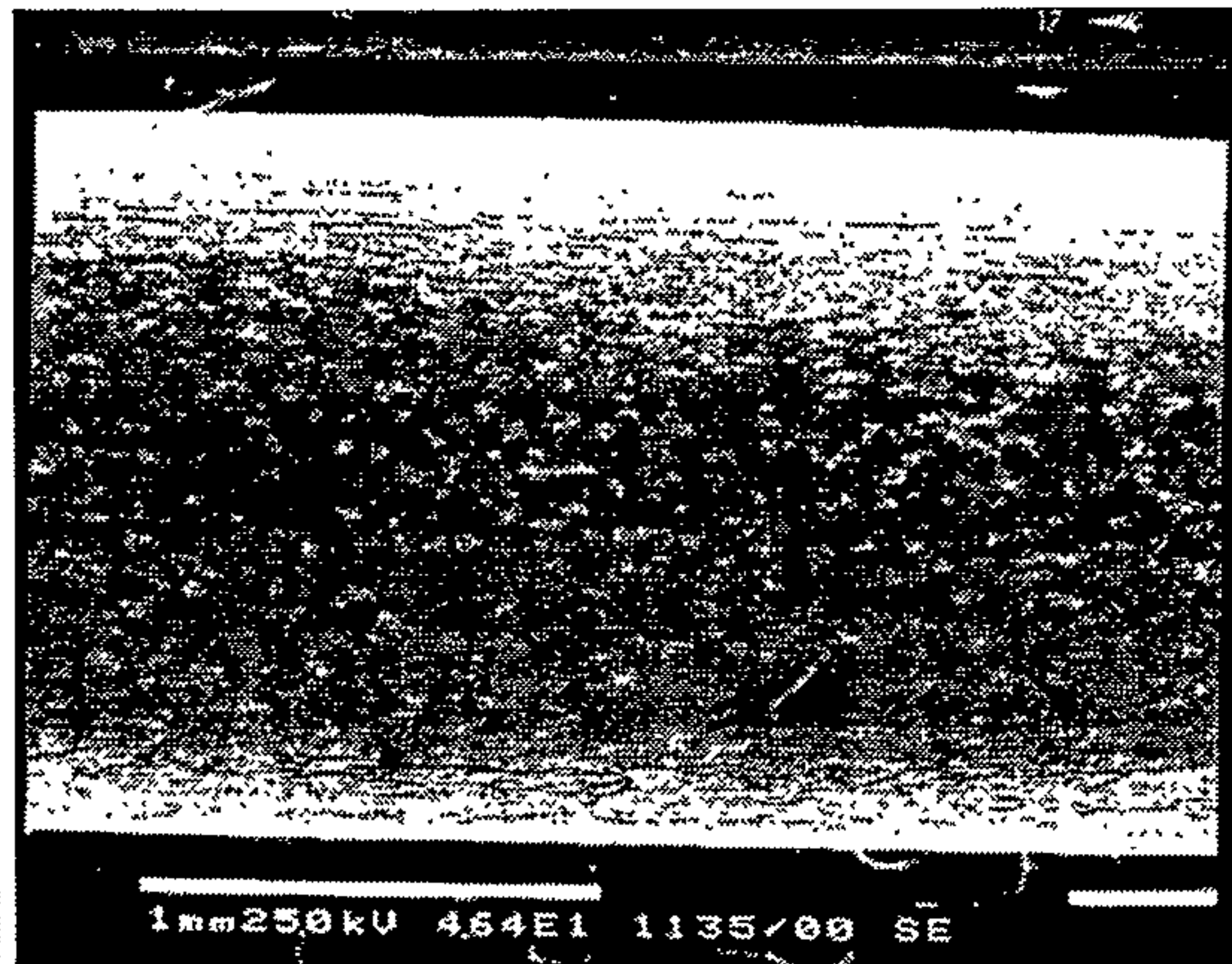


Fig.3.28. Tensile surface of 4 point bending part of WS wire.

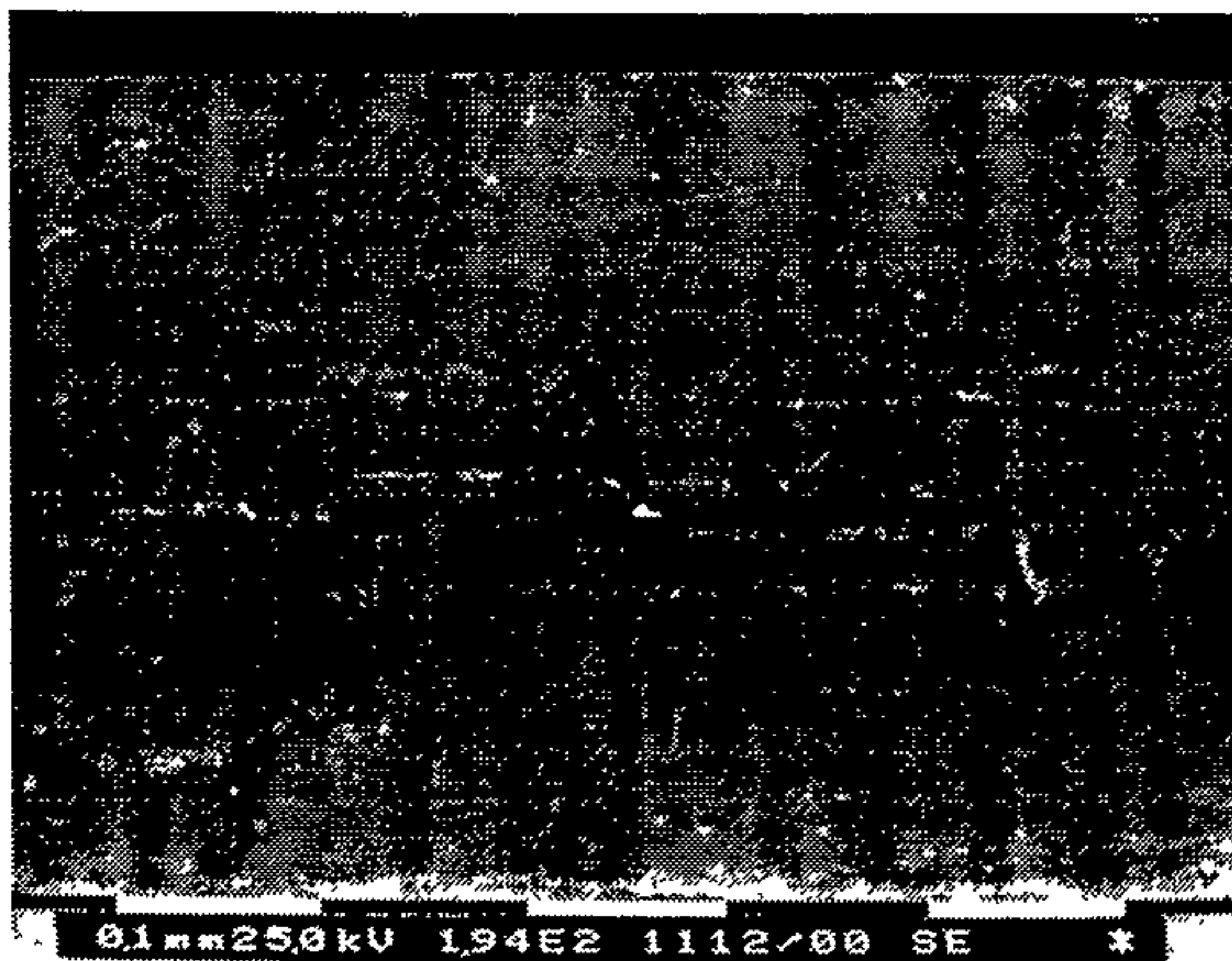


Fig.3.29. Tensile surface of 4 point bending part of GF wire.

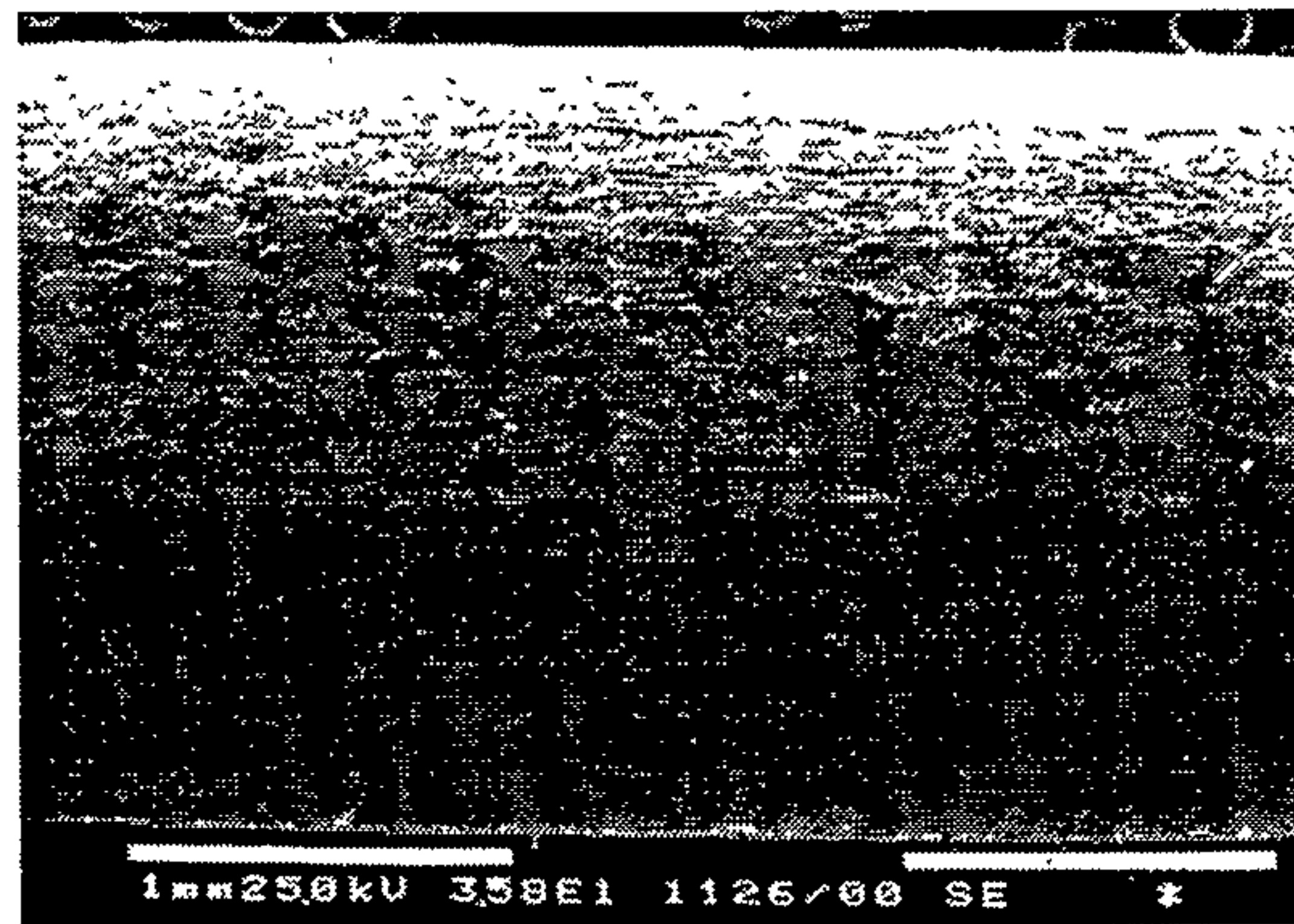


Fig.3.30. Tensile surface of 4 point bending part of GS wire.

3.5. Discussion.

3.5.1. Elastic modulus.

As discussed in standard metallurgical texts [3.20, 3.26], the elastic modulus depends primarily on the interatomic forces between the atoms. These bonds depend upon the crystal structure and the nature of the material. The method of strengthening, alloying, cold-working and heat-treatment significantly influences the microstructure but affects the value of E only slightly. Thus, for a given alloy, the modulus of elasticity is usually constant. For austenite stainless steel, the standard elastic modulus value is about 200GPa. [3.21]

However, as seen in the introduction to this chapter, various researchers have reported the elastic modulus of highly work-hardened stainless steel orthodontic wire to be lower than the standard value [3.5, 3.6, 3.7, 3.8, 3.10, 3.11] (table 3.1). To explain these lower values of E , Goldberg and Associates [14] suggested that in orthodontic wires it may be due to severe cold drawing. They believed, in the process of this cold-drawing, stress-induced changes in the lattice, residual micro-strains and preferred crystal orientation may modify the value of E .

In order to support their hypothesis, since they believed that high temperature heat-treatment could restore the cold worked structure into its original austenite stainless steel structure, they tested (in tension) the same stainless steel wires both in their as-received form and after heat-treatment. The results confirmed their hypothesis since, in the as-received wires, the value of E was found to be 20% lower than the standard whereas the value of E in the heat-treated wires was close to the standard. [3.12]

However, this result was contradicted by the flexure tests of Kusy and Dilley [3.9] who found that the value of E remained at 197-206 GPa irrespective of the amount of work-hardening.

In this study, in the tensile tests using the extensometer, the E values for the four as-received wires and the heat-treated WF wires were all in the range 190GPa-203GPa, which is consistent with the Kusy and Diller's results[3.9], but different from others[3.5, 3.6, 3.7, 3.8, 3.10, 3.11]. The results in this study are also consistent with the principle that the elastic modulus E , depends only upon the alloy and that a wire drawing process, such as cold-working or heat-treatment, should not reduce E by a factor of 20% as was found in the tests conducted by other researchers.

Although the values for E found in tension tests using the extensometer were all close to the standard value (200GPa), the values found in tensile tests not using the extensometer were in the range of 160GPa-175GPa, which is about 15% lower than the standard value. The fact that the same wires but with different methods for determining elongational strain during tensile tests (one using an extensometer across the gauge length and another one without using the extensometer but relying on the machine crosshead displacement) shows the lower E value could be due to the machine compliance error since the purpose of an extensometer is to eliminate the compliance of the machine. Therefore, the E value which is 15% lower than standard E value found in tensile tests not using the extensometer could be due to the machine compliance.

The accuracy of the value of E is very important. As described in the experimental methods section, the surface of the gages were ground down

and rounded in the extensometer we used. This ground down surface affected the accuracy of the E value. As seen in appendix 3, the inaccuracy in gauge length of the extensometer determination implies that about a 0.0003% increase should be added to the all the E values in the tensile tests results. This correction of the E value eliminates the experimental error for the extensometer we used, but does not change the E value much (only about 3%). Thus, the above discussion about E value above is still valid.

In the flexure test (both 3 point and 4 point bending tests), the values for E were all about 170GPa, which is about 15% lower than the standard value. This lower E value obtained from the flexure tests could mainly due to the compliance of the machine since an extensometer cannot be used in bending tests. To measure the machine compliance in the flexure tests condition, a suggestion was that a flexure test on a glass rod be used to measure machine compliance. Unfortunately, the result was not conclusive (c.f. appendix 1). Another possible effect on the lower E value was large deflection effect. Again, this could not be proved to be the cause (c.f. appendix 2).

Since the values of E in bending tests involve machine compliance and other effects, less confidence is possible in the values of E derived from the bending test as compared to those derived from tensile tests using the extensometer.

3.5.2. Yield strength

3.5.2.1. The comparison of yield strength between the four wires.

The 0.1% YS measurement represents the elastic limit of wires. The tension test results given above suggest that the ordering from highest to lowest in terms of 0.1%YS is:

$$WF > GF > WS > GS.$$

Since all of the wires have the same composition, the difference in strength can only be due to their microstructure. As we discussed in chapter 2, several microstructural factors can affect the 0.1% yield strength of the wires:

- grain size;
- twinning density;
- the amount of martensite phase; and
- small angle boundaries.

The detailed discussion about the relationship between microstructure factors and yield strength will be discussed later in chapter 5. In the following section, this discussion is summarised so that the tensile 0.1% yield strength results can be interpreted.

3.5.2.1.1 Grain size:

In the results from the TEM experiments in Chapter 5, it will be seen that the ordering of grains or subgrains size from largest to smallest is

$$GS > WS > GF > WF.$$

This ordering is precisely the opposite to that of the tensile 0.1% yield strengths found above. A finer-grained material is harder and stronger than one which is coarse-grained. The reason is that a smaller grain size means a greater total grain boundary area to impede dislocation motion. Smaller grain size also increases the dislocation density, thereby significantly contribute to the work-hardening of the structure. [3.28; 3.29; 3.30].

The TEM grain size and related yield strength results are consistent with the Hall-Petch equation which expresses the relationship between strength and grain size. [3.31; 3.28] Details of these are described in the discussion part of chapter 5.

3.5.2.1.2. Twinning density.

As well as the above grain size factor, the density of the twinning in the wires also can effect yield strength.

In the TEM experiments, a high density band twin structure was observed to exist in WS, WF and GF wires and a low density band twin structure exists in GS wire. Thus, the WF, WS and GF wires have higher yield strength than GS wires, as is confirmed by the tensile test results.

Heavy deformation occurs during the wire manufacturing process. The concentration of stress results in the wires being unable to continue to develop by drawing slip and so a form of twinning occurs in order to continue the deformation. The shear stress for forming twinning is higher than for slip. [3.32; 3.29].

3.5.2.1.3. The amount of martensite

From the XRD experiments in Chapter 4, we obtained the following volume percentages of martensite phase:

GF: 82%; WF: 74%; GS: 70%; WS: 56%.

Martensite transformation in steel is one of the most common strengthening processes used for engineering materials. The reason for martensite strength of materials is that martensite has many strong barriers to dislocation motion. These barriers are provided by a fine twin structure or a high dislocation density. Another reason for martensite strengthening is the carbon atom clustering and the strong binding set up between dislocation and the carbon atoms. [3.28]

However, the yield strength of these four wires is not exactly proportional to the amount of martensite phase. The reason is because yield strength is affected by the combination of factors mentioned above, not only the amount of martensitic phase.

3.5.2.1.4. Small Angle Boundaries

TEM results show small angle boundaries exist in the WS, WF and GF wires. As we mentioned in chapter 2, small angle boundaries actually significantly strengthen the wires [3.28; 3.33; 3.34]. This is another reason why yield strength in these three wires is much higher than in the GS wires.

3.5.2.2. The comparison of yield strength between the type of tests for same wires.

For the same wires, different 0.1% yield strength value were found in the 3 point bending tests, the 4 point bending tests and the tensile tests. For each type of wire, the values of yield strength was always in the order:

three point bending > four point bending > tensile test.

These results are different from the test results in Drake et. al. [3.8]. In their paper, they found that, for stainless steel orthodontic wire, the bending values of 0.1% yield strength were roughly comparable with values from tension tests. But later, Asgharnia et. al [3.7]. found the value of 0.1% YS to be significantly higher in bending tests than in tensile tests. Asgharnia's work corresponds with the results in this study.

The difference in 0.1% YS values between the tests can be explained by the different nature of the two modes of deformation. During bending tests, the outermost portion of the specimen cross section initially undergoes permanent deformation, while the remainder of the specimen is still within the elastic range. In contrast, during the tension tests the entire cross section of the wires is assumed to initially undergo permanent deformation in a uniform manner. Moreover, the nature of 3-point bending test is that a maximum stress is concentrated on to a single point. In 4-point bending tests, stress is concentrated uniformly between the two ends at which force is applied. In the tensile test, a uniform stress is applied across the entire test length of the wire.

The highest level of yield stress measured in the 3-point test is greater than the highest level of stress in the 4-point test. The stress of the 4-point test is, however, higher than the stress applied in the tensile test. Due to the different stress distributions of the different tests, the value of 0.1%YS in the 3-point bending test is the highest, and the value in the tensile test is the lowest.

3.5.3. Ductility

3.5.3.1. Tensile test.

In the tension tests, the elongation at fracture point represents the ductility of the wires. The above tension tests indicate that GS wires is ductile whereas the other three wires are brittle. These results can be confirmed by SEM micrographs which provide information about both the macro-mechanisms and the micro-mechanisms of the wire fracture.

The pictures (fig.15-fig.17) of the WF, WS and GF wires show “cup & cone” fractures whereas the pictures (fig.18) of the GS wire shows a semi cup & cone moderate fracture. Hence, the macro-mechanism of the fractures imply that GS wires are more ductile than the others. This result agrees with the tensile test results shown above.

The SEM figures (fig.19,20,21 & 22) are the fracture surfaces of WF, WS, GF and GS wires respectively. These figures provide information about the micro-mechanism of the wire fractures. From all these SEM micrographs, only GS wire shows necking, this further confirms that GS wires are more ductile than the other three wires.

3.5.3.2. Flexure test.

In the 3 point and 4 point bending tests, the SEM micrographs at the surface of the tension side (fig.23-30,) suggest that only Wilcock's wires show cracks on the tension surface in both the 3 & 4 point bending tests. This implies that Wilcock's wires are much more brittle than the other three wires. This result is consistent with the tensile results.

For Wilcock wires, the fact that cracks appear on the tension surface but do not extend on to the side surface is because the tension stress is at maximum level at the surface and gradually reduces to zero as it approaches the middle of the wires (the neutral axis of the wire).

3.6. Conclusions

The following results can be drawn from this study:

- The elasticity modulus measured in the tensile tests on the four as-received wires and the set of annealed WF wires were all approximately 200GPa. This result is contrary to the results of tests by other researchers but consistent with the principle that the elastic modulus, E , depends only upon the alloy and wire drawing processes, such as cold-working or heat-treatment, should not reduce the value of E .
- The elasticity modulus measured in the tensile tests without using the extensometer, 3 point bending and 4 point bending tests were all about 170GPa, which is 15% lower than the value in tensile test with using the extensometer and the standard value. This difference may be due to the machine compliance error since an extensometer was not used.

- The tension test results suggest that the 0.1%YS are different in these four same composition wires. This variation in strength would seem to be due to the differences in microstructure found in the TEM and XRD chapters, particularly the different grain sizes given that the results for yield strength and grain size exactly fit the Hall-Petch formula.(taken up in chapter 5)
- For the same wire, different 0.1% yield strength values were found using 3 point bending tests, 4 point bending tests and tensile tests. These differences can be explained by the different nature of the deformation modes.
- The tensile test and SEM micrographs suggest that the GS wires are considerably more ductile than the other three wires and that the WF wires are the most brittle.

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Appendix 3.1. The measurement of machine compliance using glass rods under flexure test conditions.

To analyse machine compliance and thereby to determine the value of E , a 3mm diameter glass rod was tested under the same conditions as the 3 point bending tests on the GF wires. Since glass is an elastic-brittle material which has no microstructure, if the E value is lower than the standard value (68GPa), the lower amount should be due to the machine compliance. In this test the deflection of the specimen was small and there appeared to be no slippage of the specimen on the loading points.

In fig.3.9A, the (1) line is the load-stroke curve obtained from the 3 point bending glass rod test. The E value is about 52GPa. Line (2) is the line which have the standard glass E value of 68GPa. By using line (2) minus line (1), line (3) is obtained which represent the compliance of the machine.

Applying this machine compliance then corrects the force-displacement curve for the GF wires 3 point bending tests. In fig 3.9B, line (4') is the machine compliance line obtained from the above glass rod test (see fig.3.9A). Line (1') is the line have E value of 150GPa which obtained from the 3 point bending test of GF wires. By putting lines (1') and (4') together, line (2') is obtained which represents the E value after correcting for the machine compliance. This E value after correcting for the machine compliance is only about 155GPa which is far from the standard GF wires E value (200GPa) (see line 3'). Therefore, the glass rod bending test does not assist in determining whether machine compliance is the cause of the difference in E values in 3 point bending tests.

Load stroke curve for glass

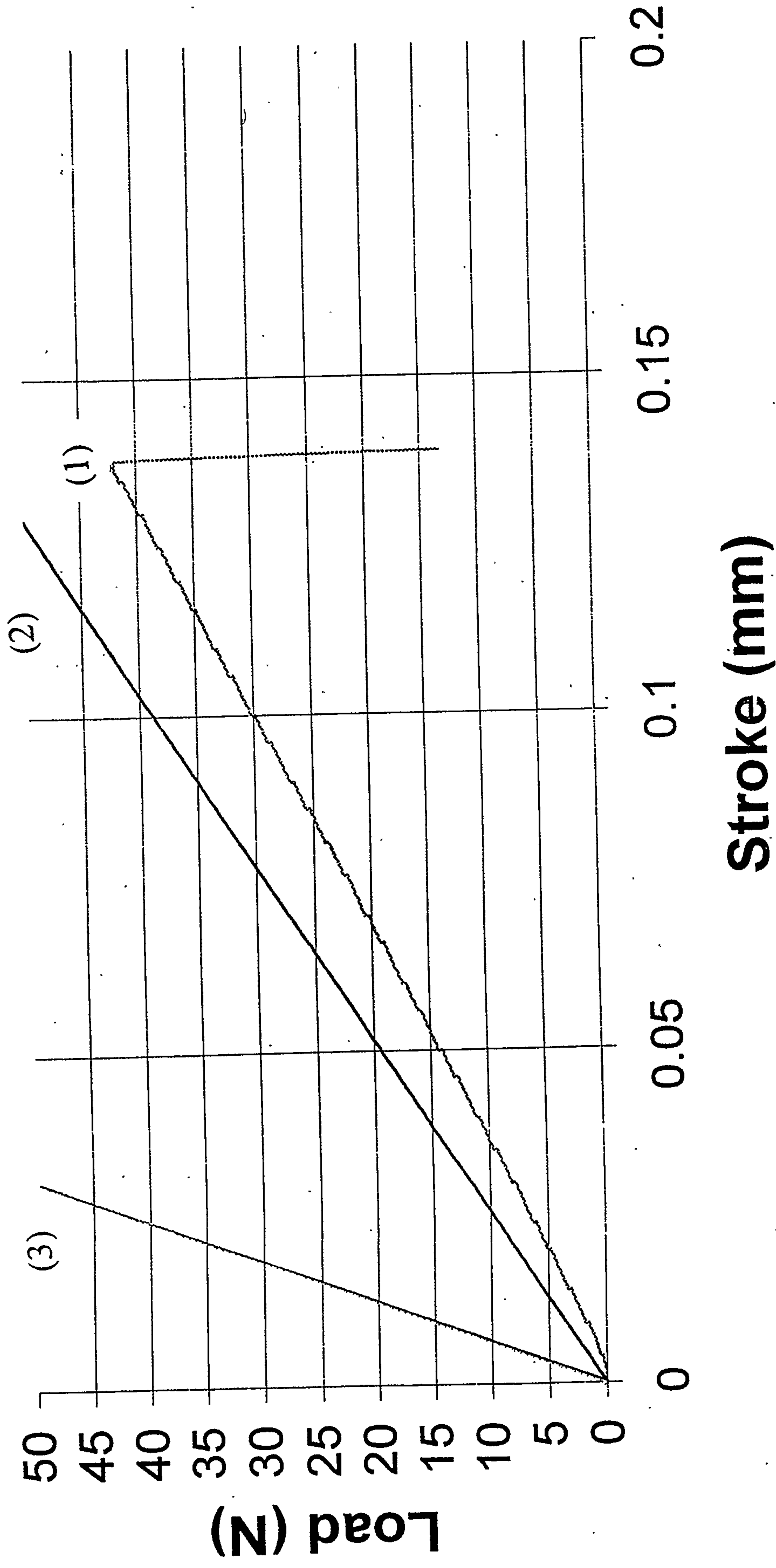


Fig. 3.9A

Load stroke curve foe GF wire.

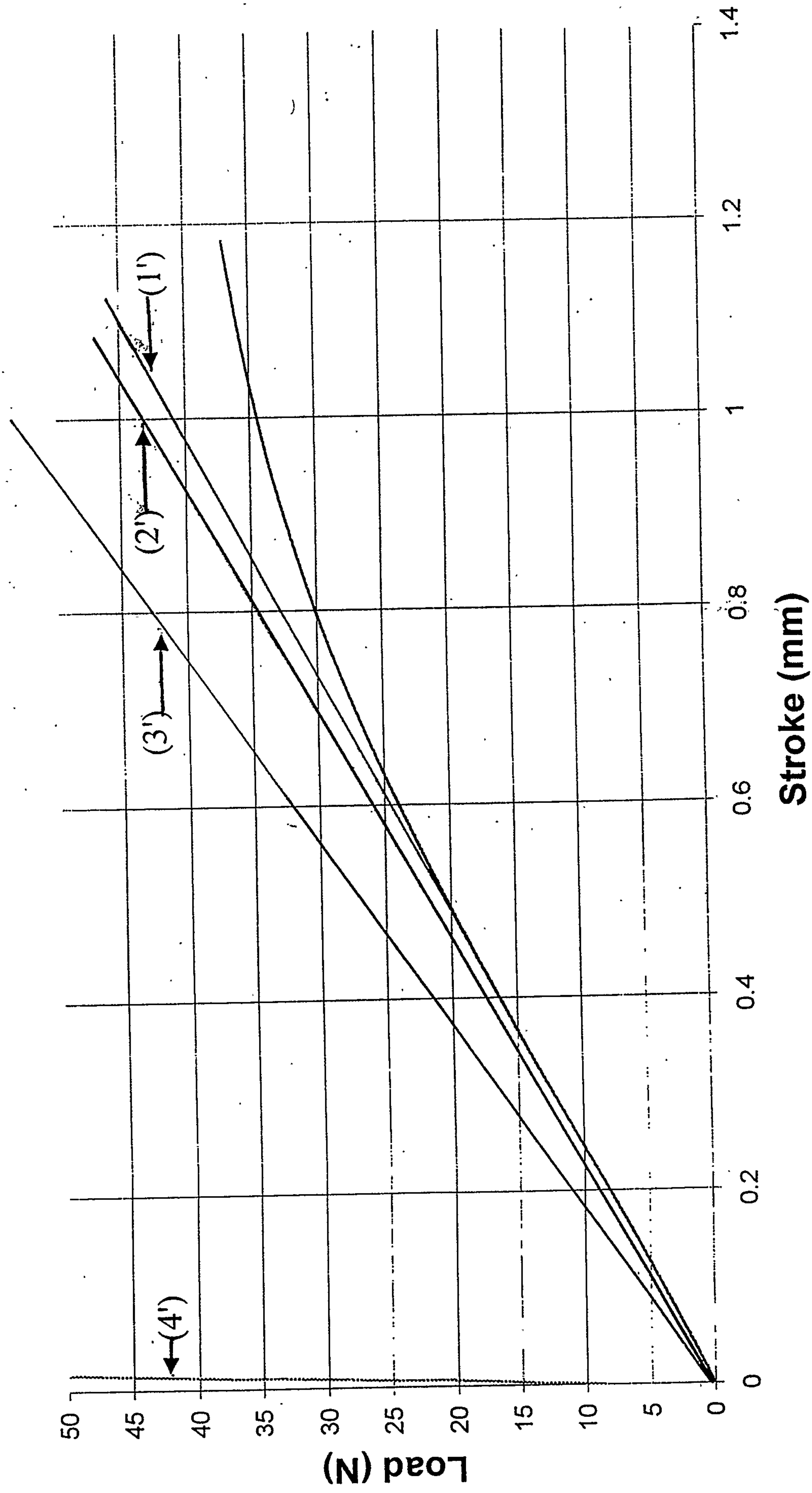


Fig. 3.9B

Appendix 3.2 : Examination of the possible cause of a lower E value due to large deflection on the gauges during the flexure test process

For bending tests, the formulae for calculating stress and strain are based on small deflection conditions. However, some of the wire bending tests generate significantly large deflections. Hence, we need to correct the E value for the large deflection condition. From the figure 3A which is adopted from the Timoshenko text book "Mechanics of Materials" [3.27], we see that $\delta v/L < 3$ is the condition for small deflection theory and $\delta v/L > 3$ is the condition for large deflection theory. δv is the vertical displacement of the beam and L is the initial length. (see fig.3.10). From table 3.A, we see that, in the 3 point bending tests, large deflection theory applies only to Wf wires and in the 4 point bending, large deflection theory applies to WF WS and GF wires.

Since we know the value of $\delta v/L$, from fig.3A, we can obtain the PL^2/EI value for a small deflection (A) and PL^2/EI value for a large deflection(B). (P is the load; E is elastic modulus and I is the bending moment.) Therefore, $PL^2/E_{\text{small deflection}} I = A$ and $PL^2/E_{\text{large deflection}} I = B$, Thus, $E_{\text{large deflection}} = (A/B) * E_{\text{small deflection}}$.

The E values after the correcting for a large deflection in WF wires in 3 point bending and in WF, WS and GF wires in 4 point bending are given in table 3.B. These results shows that the large deflection correction lead to a decrease in the value of E calculation.

Table 3A

3 point bending					
Wire	Diameter (D) (mm)	Strain (ϵ)	Span length (L')	δv	$\delta v/L'$
WF	0.52	0.06	7	3.77	0.54
WS	1.62	0.035	7	0.71	0.11
GF	0.88	0.05	7	1.86	0.27
GS	2.18	0.026	7	0.39	0.06
4 point bending					
Wire	Diameter (D) (mm)	Strain (ϵ)	Span length (L')	δv	$\delta v/L'$
WF	0.52	0.042	4.5	2.908	0.646
WS	1.62	0.050	13.8	6.012	0.437
GF	0.88	0.045	4.5	1.840	0.409
GS	2.18	0.040	13.8	3.574	0.259

Table 3B

3 point bending					
Wire	Diameter (mm)	E (GPa) (small deflection)	PL^2/EI	PL^2/EI	E'
WF	0.52	168	1.5	2.4	105
4 point bending					
Wire	Diameter (mm)	E (GPa) (small deflection)	PL^2/EI	PL^2/EI	E'
WF	0.52	160	2	3.6	89
WS	1.62	160	1.4	1.95	115
GF	0.88	160	1.2	1.6	120

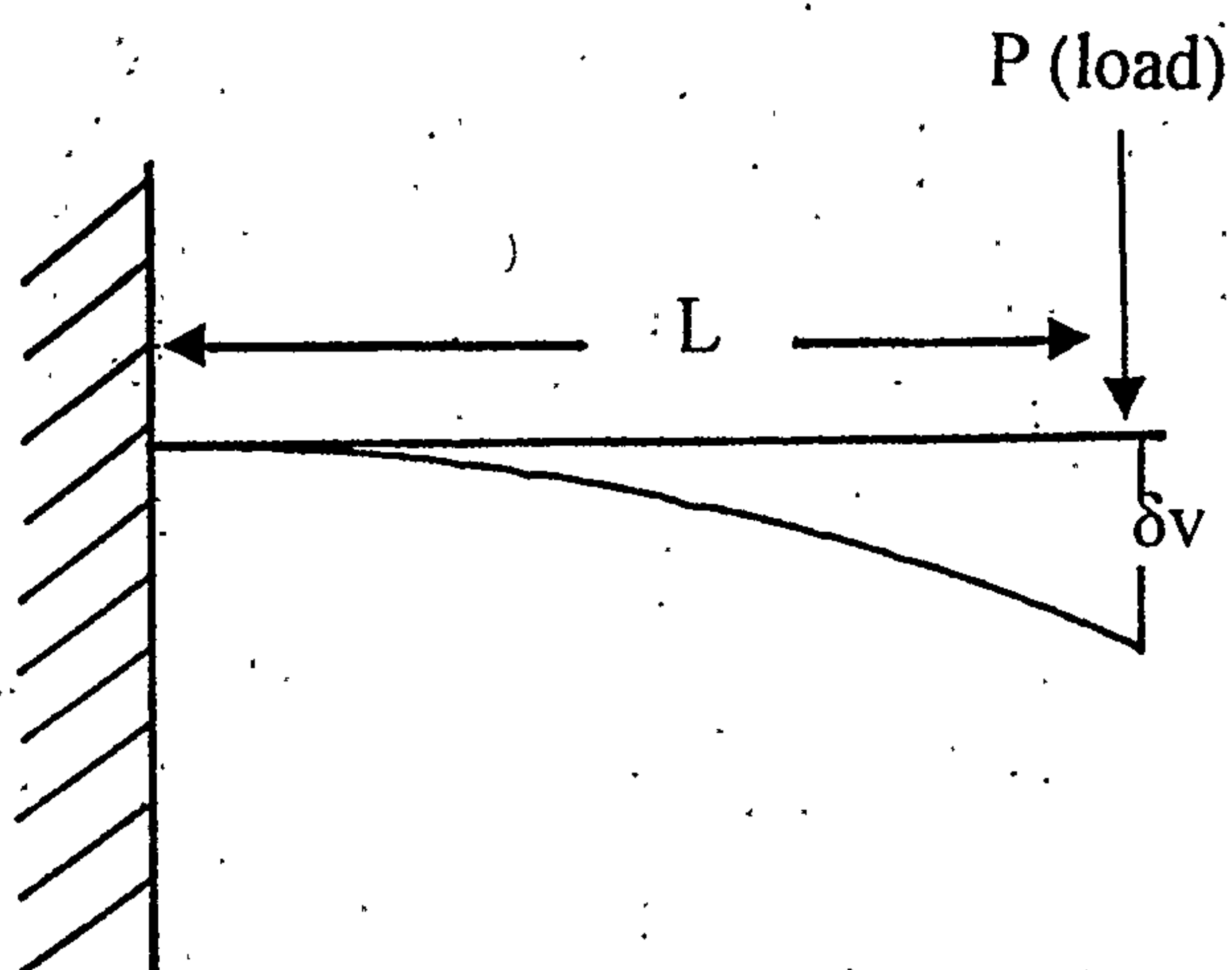


Fig. 3.10

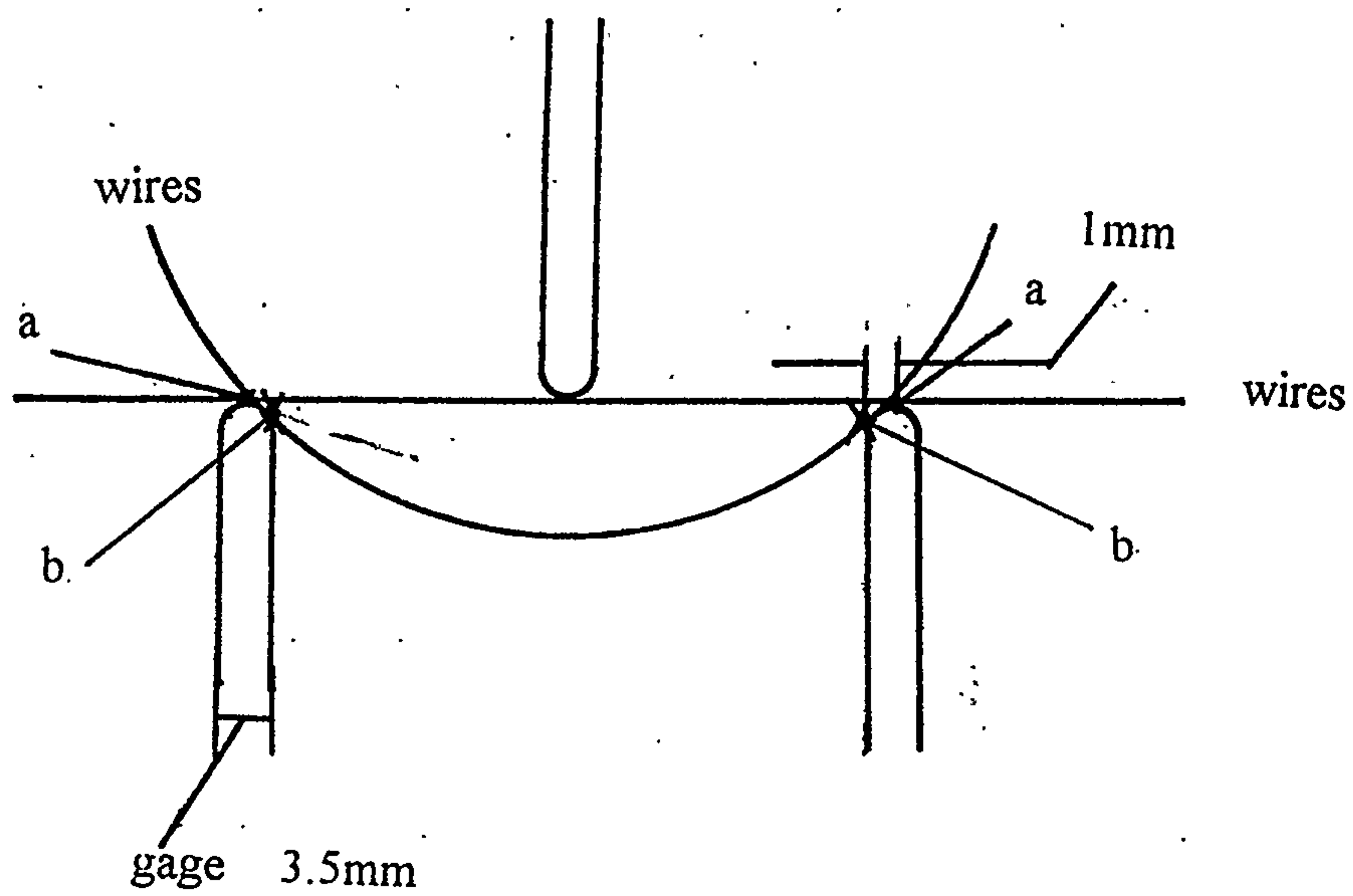
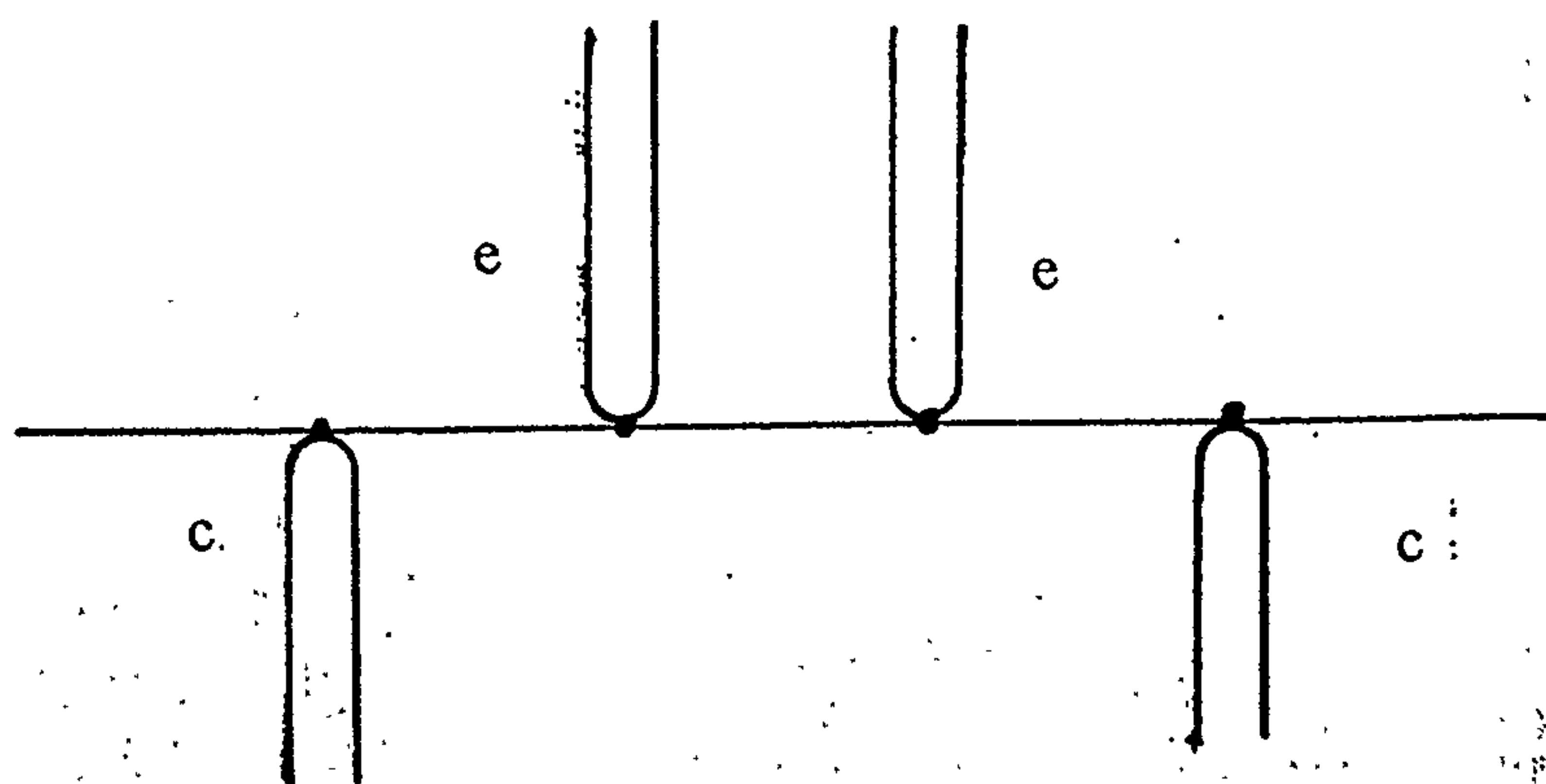
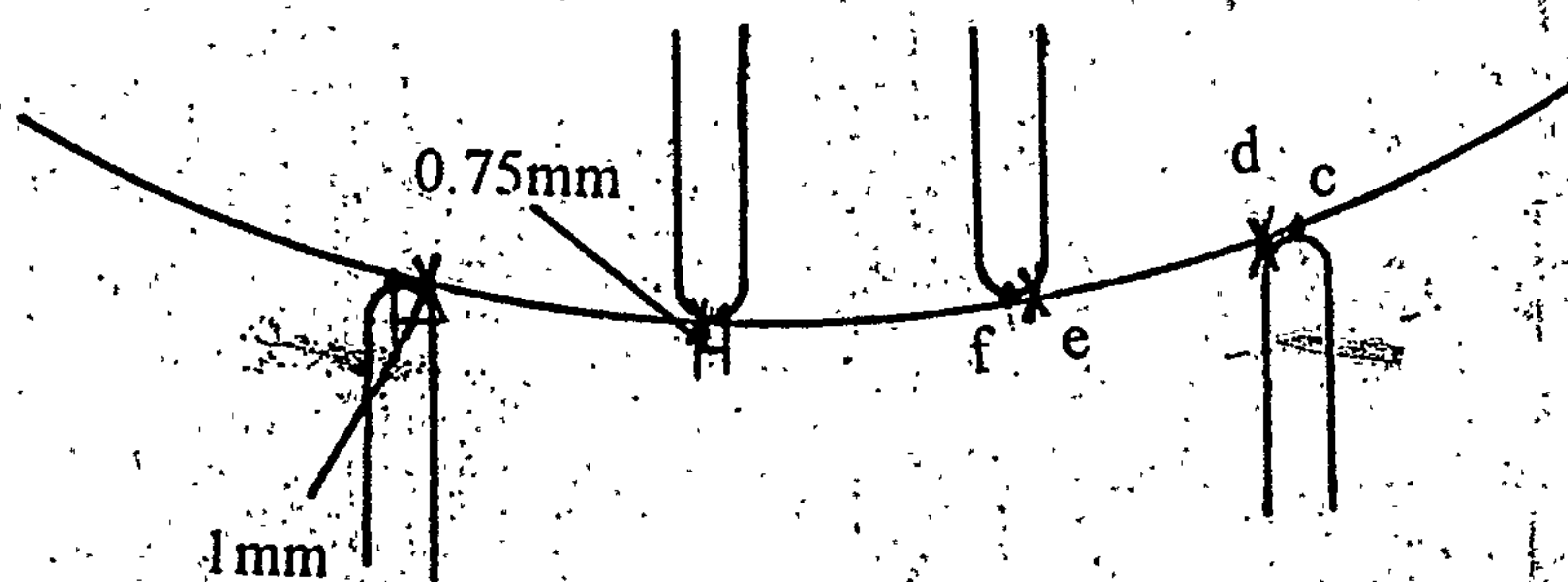


Fig. 3.11



(a)



(b)

Fig. 3.12

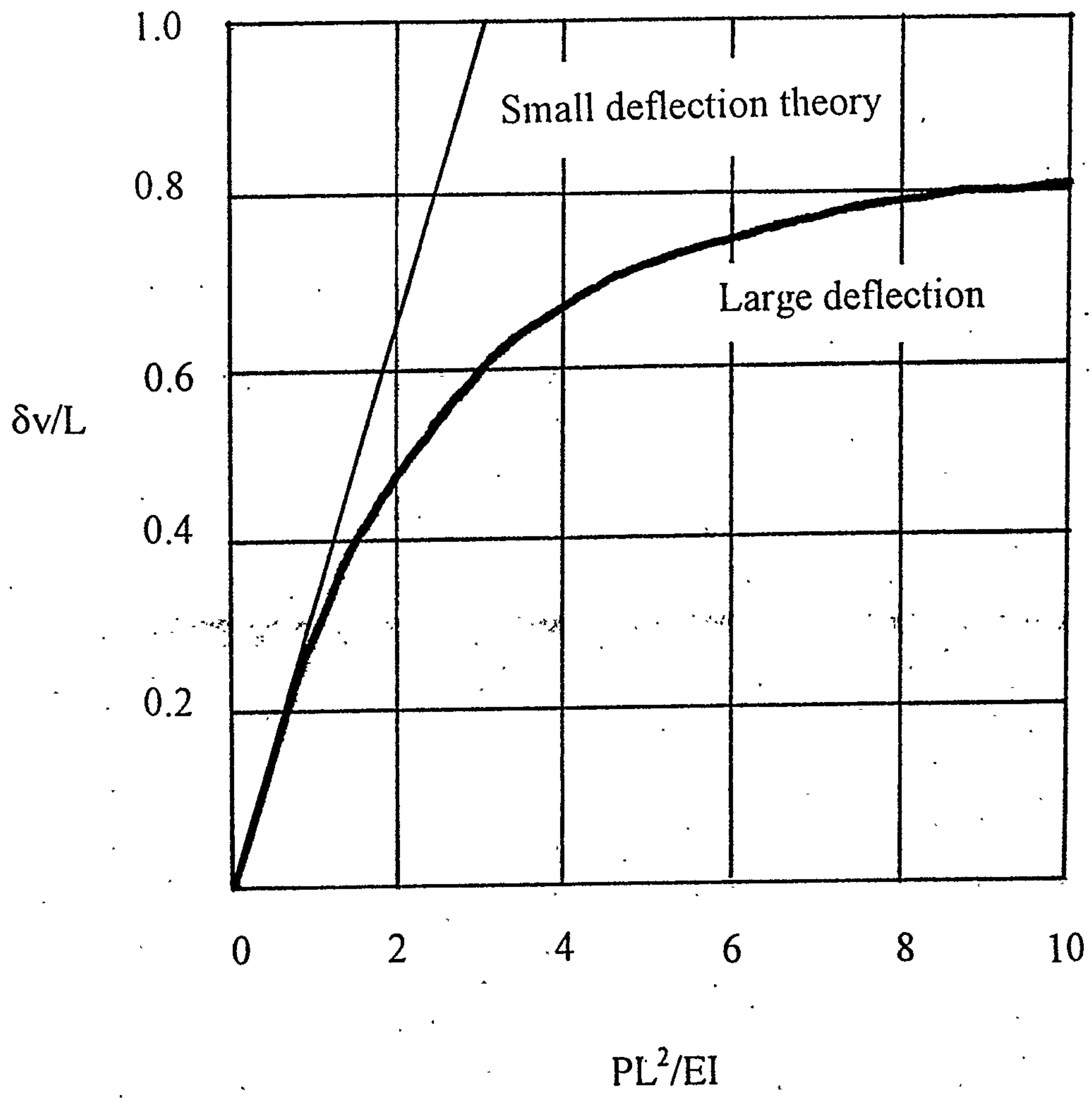


Fig. 3A

Appendix 3.3: The derivation of the effect of the ground down surface in the extensometer upon the accuracy of the value of the elastic modulus.

Fig.31 illustrates our extensometer. The shaded area represents the ground surface. From the diagram, it can be seen that the ground surface can reduce the gage length from 10mm to about 9.7mm. The value of strain calculated by the computer analysis software is based on a 10mm gage length, but the actual value of strain should be calculated based upon a 9.7mm gage length. The percentage error of strain is as follows:

$$\%error = \frac{\frac{\Delta l}{10} - \frac{\Delta l}{9.7}}{\frac{\Delta l}{9.7}} = 0.0003\%$$

The above % error is constant unless the gage length changes.

So, the correct strain value (ϵ_c) should be:

$$\frac{\epsilon_v - \epsilon_c}{\epsilon_v} = -3\%$$

$$\epsilon_c = \epsilon_v(1 + 0.0003)$$

where: ϵ_v is the strain value read from the computer.

Thus, the correct E value should be:

$$E_c = \frac{\sigma}{\epsilon_c} = \frac{E}{1.0003}$$

Where:

E is the value read from the computer, and

E_c is the correct elastic modulus value.

Thus, all the E value of tensile tests and figures should be divided by about 1.0003 to obtain the correct value of elastic modulus. Therefore, all the tensile elastic modulus value in the Appendix 2 should increase about 0.0003%.

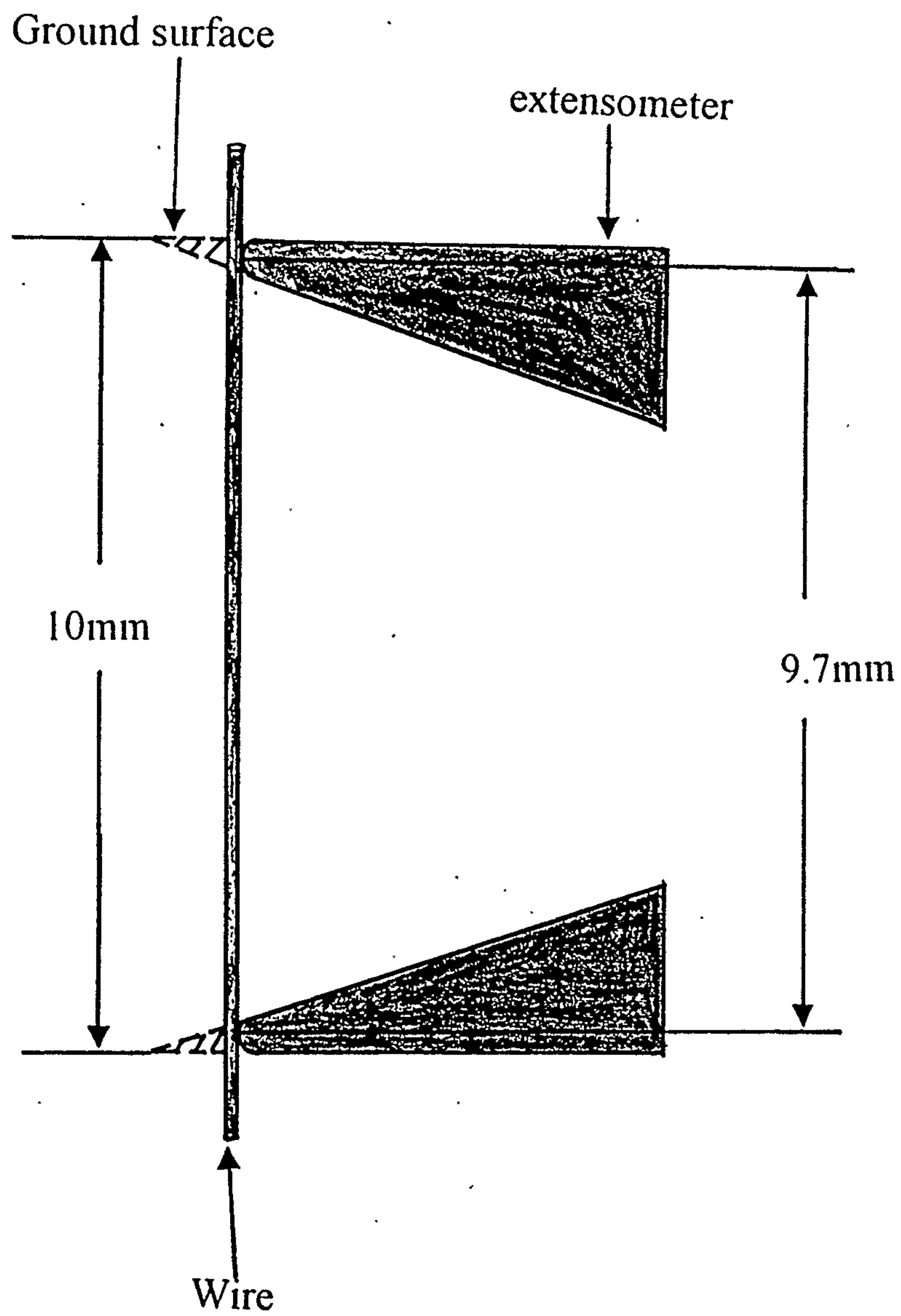


Fig 3.31 Extensometer with ground surface.

Chapter. 4 XRD investigation of GS,GF,WS and WF wires.

4.1 Introduction:

The properties of AISI 302 type stainless steel orthodontic wires are governed primarily by their microstructure and their chemical composition. Because the alloy, AISI 302, has a standard composition, the different mechanical properties of different orthodontic wires heavily depend on the effect of the microstructure and one of the main factors controlling this is the forming process (see 2.6). Type 302 orthodontic wires are produced by a cold drawing process combined with intermediate annealing heat treatments. Although the basic atomic arrangement of 302 stainless steel is face centred cubic austenite, partial transformation to alpha or epsilon martensite occurs during the drawing of the wire owing to the large deformation associated with this process[4.1, 4.2]. One of the benefits of the stress-induced transformation of austenite to martensite is the increase in strength of orthodontic wires. For clinical applications, therefore, it is important to be able to measure and control the relative proportions of austenite and martensite in order to optimise and identify the conditions for high strength without loss of ductility. During the drawing of the wire, another distinct phenomenon introduced is the texture. A strong preferred orientation will result in an anisotropy in mechanical properties. This can result in uneven response of the material during forming.[4.3]

Transmission Electron Microscopy (TEM) is ill-suited for quantifying austenite and martensite phases from TEM images because both phases are twin structures of almost identical appearance in the wires. Furthermore, even though the TEM diffraction pattern from each phase is different, it is

difficult to use diffraction patterns to accurately quantify these phases. Moreover, the TEM technique only shows a very small area of the wires and the results are not always representative of the whole wire. XRD can overcome these disadvantages since it clearly resolves the austenite and martensite phases as different diffraction peaks. With the XRD technique, the texture of austenite and martensite in the wires can be quantified separately.

In this chapter the phases and their texture in orthodontic wires are quantified by X-ray diffraction (XRD). The influence of texture and phases to the mechanical property will be discussed in chapter 5.

XRD is a well established technique for quantifying the martensite and austenite [4.4, 4.5] because the peaks from these phases are easily resolved even in heavily deformed and fined grained materials used in the present investigation (see fig.4.1). Provided the crystallites in the material are randomly oriented or nearly randomly orientated, it is a relatively simple matter to quantify the phase content of the wires through an analysis of the integrated intensities of the diffraction peaks. However, orthodontic wires generally have a strong texture and the conventional quantitative XRD intensity relations based on randomly oriented crystals are invalid. Accordingly, an experiment was designed to make the orientated wire more comparable with the powder pattern by mounting the same area of wires at three different angles (fig.4.2): level (0°); upright (90°); and at an angle of 45° .

A spinning specimen stage was also used. From this experiment, the volume percentage of martensite and austenite phases in these wires was expected to

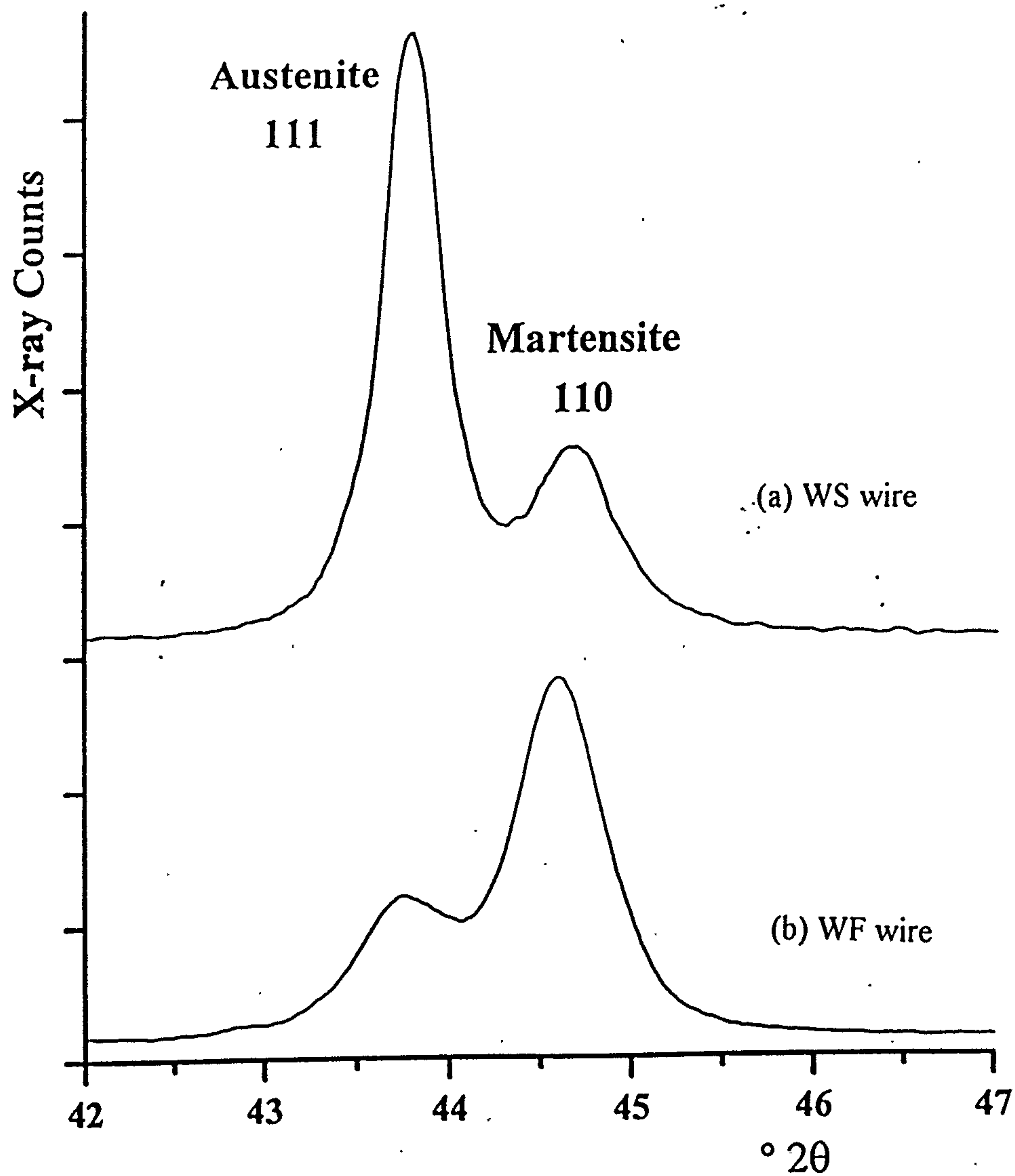


Fig.4.1 111 Austenite and 110 martensite profiles from two orthodontic wires, (a) WS($D = 1.62$ mm) and (b) WF ($D = 0.52$ mm). A large proportion of the austenite is transformed into martensite during the extrusion process.

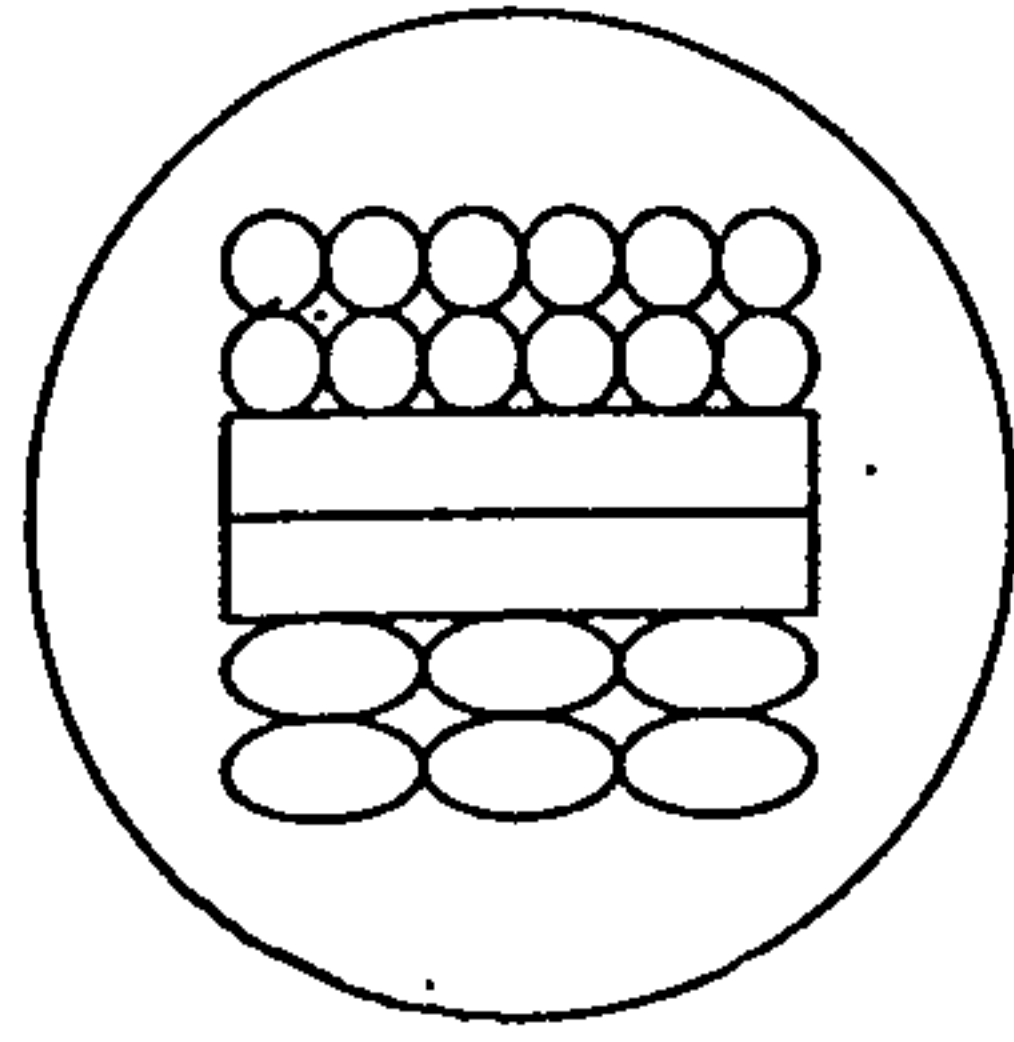


Fig.4.2 Types of specimens prepared for XRD analysis in experiment 1 to measure the martensite/austenite content in orthodontic stainless steels.

be obtained. However, it was found that, despite these steps, it is difficult to quantify the volume of the phases. At the end, for accurate quantification, a second experiment was designed, involving the development of a method for determining the austenite and martensite phase content even when a strong texture exists in wires.

4.2. Quasi-Powder Approach.

4.2.1. Materials, method and measurement theory.

Two different brands of orthodontic wire, type W wires with diameters of 0.52 (WF) mm and 1.62 (WS) mm and type G wires with diameters of 0.88 mm (GF) and 2.18 mm (GS) were used.

All the XRD data were collected by reflection diffraction using a conventional Siemens D5000 diffractometer with a graphite diffracted beam monochromator.

The XRD spectrum shows that these four wires contain only the γ -austenitic and α -martensitic phases (Figs.4.3, 4.4, 4.5 & 4.6). Other phases that are known to occur in orthodontic wires, such as ϵ -martensite, δ -ferrite (δ -Fe), chromium carbide (Cr_{23}C_6 , Cr_7C_3) and iron carbide (Fe_3C), [4.1, 4.3, 4.4] were not observed.

According to [4.4], the integrated intensities of the martensite and austenite diffraction lines, I_M^{hkl} and I_A^{HKL} , are given in terms of the volume fractions V_M and V_A ($=1 - V_M$) of each phase by

$$I_M^{hkl} = \frac{CV_M m_{hkl} LP(\theta_M) F_M^2 e^{-B_M/2d_M^2}}{v_M^2} \quad \text{and} \quad I_A^{HKL} = \frac{CV_A m_{HKL} LP(\theta_A) F_A^2 e^{-B_A/2d_A^2}}{v_A^2} \quad [4.1]$$

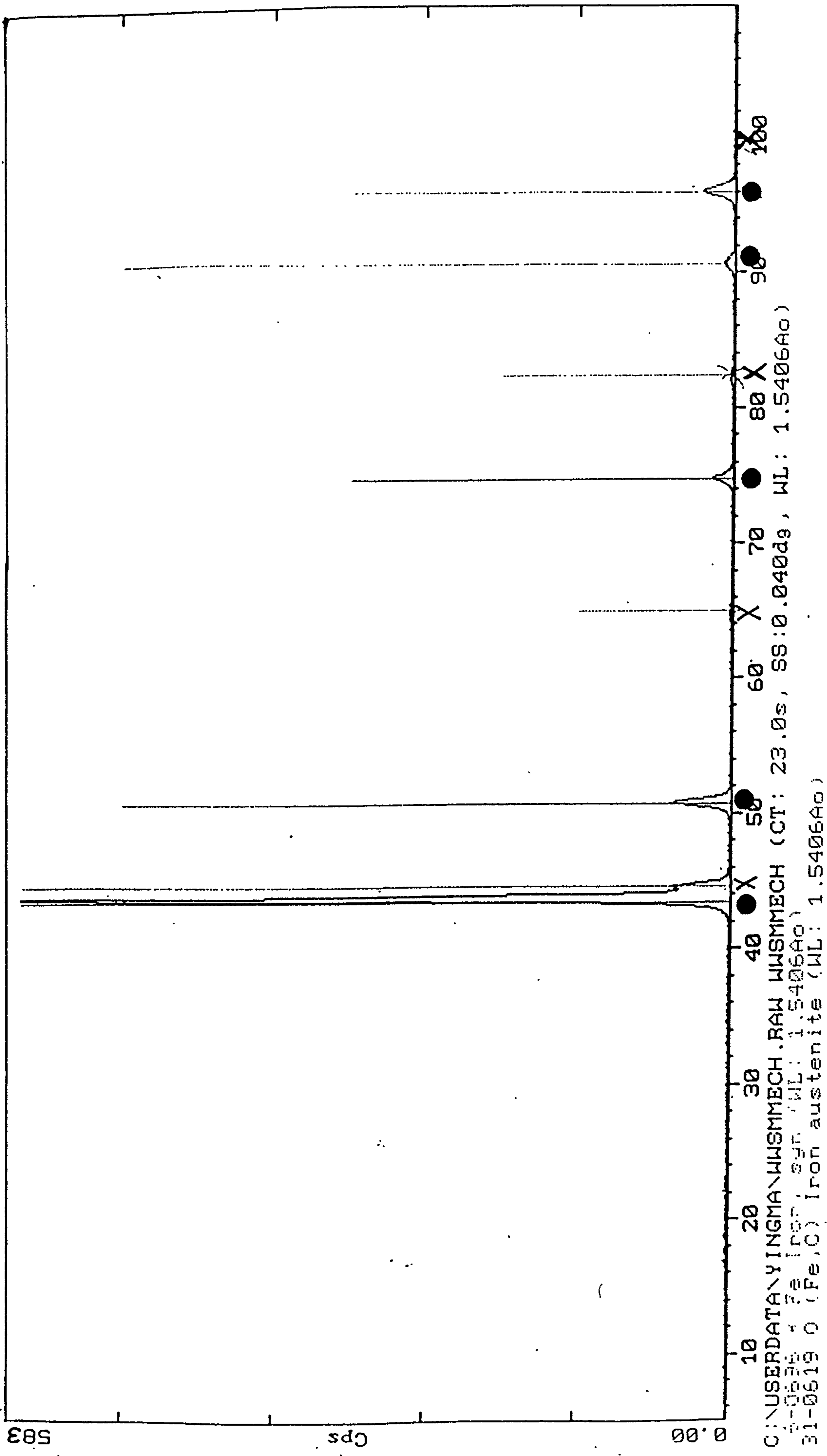


Fig. 4.3 XRD spectrum from WS wires. (●" represent the austenitic phase peak; "X" represent the martensitic phase peak.)

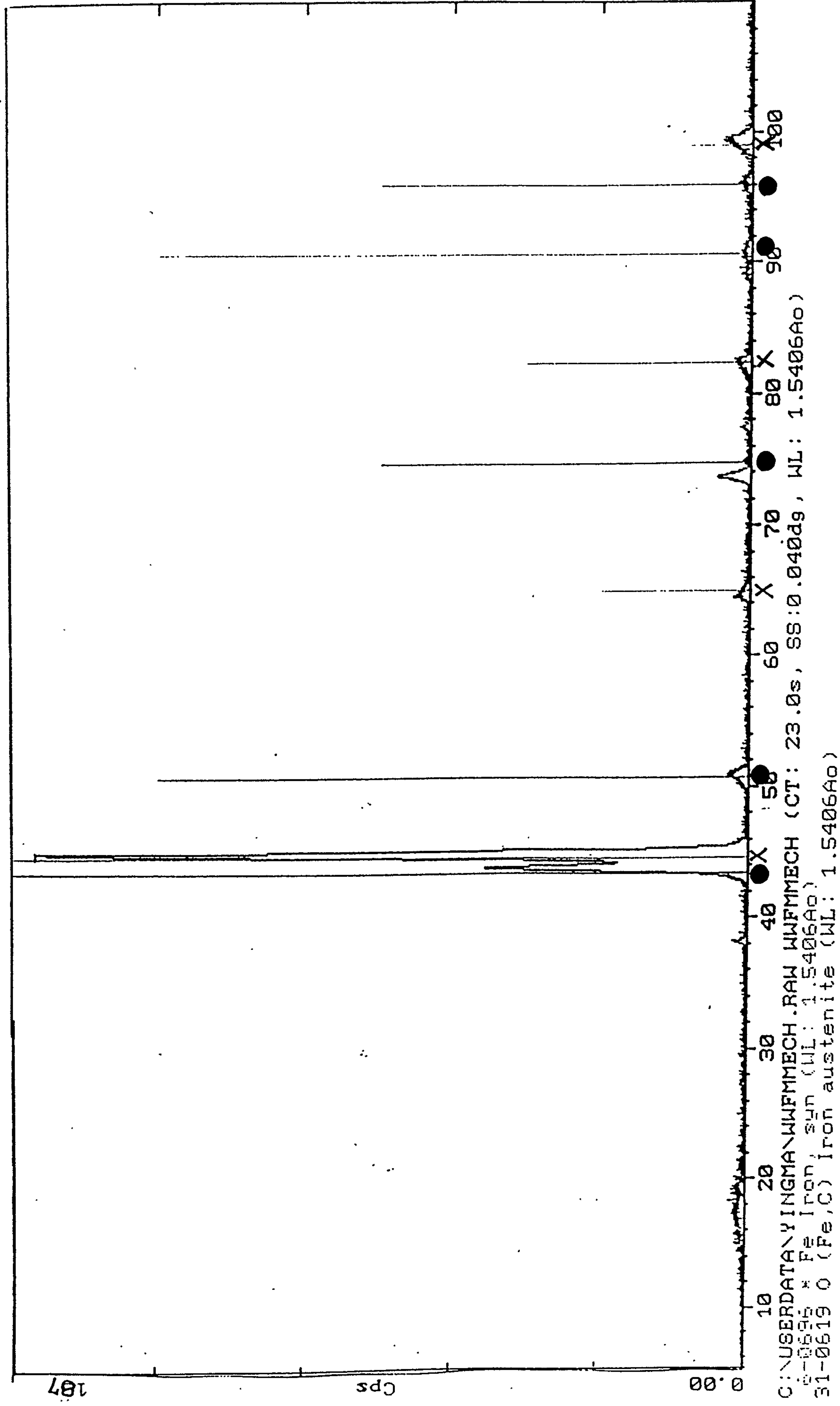


Fig. 4.4 XRD spectrum from WF wires. (● represent the austenitic phase peak; "X" represent the martensitic phase peak.)

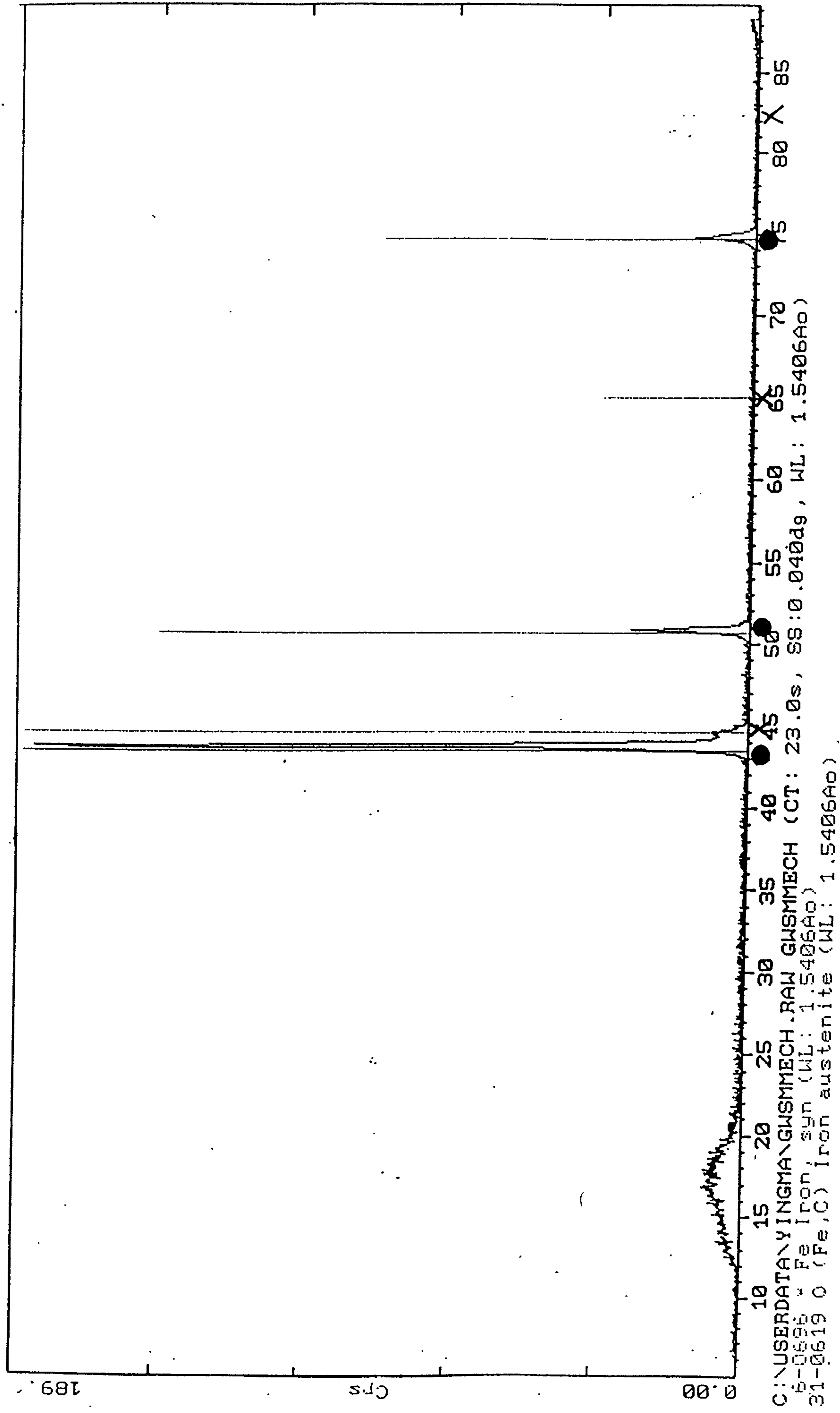


Fig. 4.5 XRD spectrum from GS wires. (● represent the austenitic phase peak; "X" represent the martensitic phase peak.)

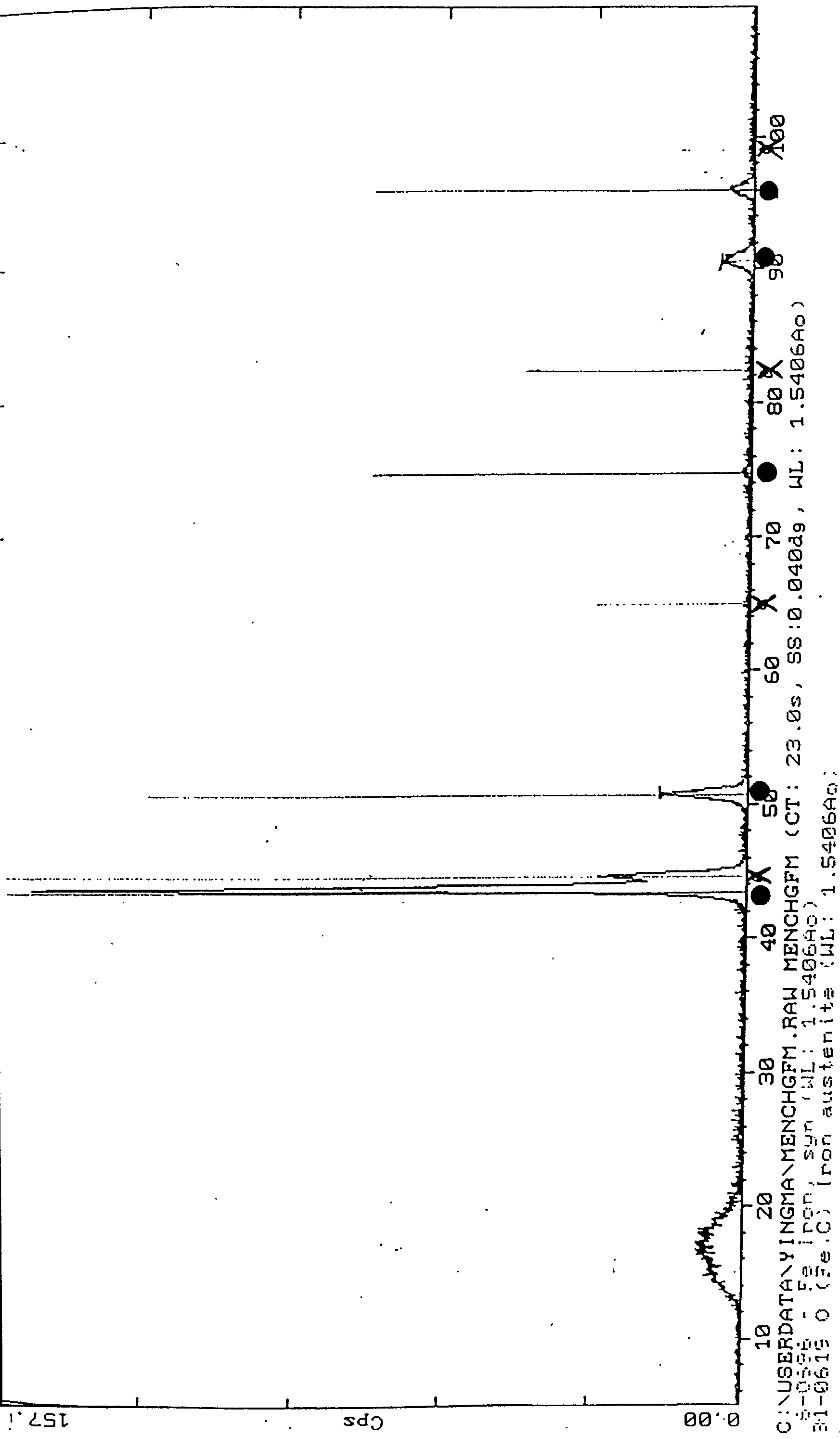


Fig. 4.6 XRD spectrum from GF wires. (● represent the austenitic phase peak; "X" represent the martensitic phase peak.)

where C is a constant embodying the dimensions of the diffractometer, the incident beam power and the attenuation coefficient μ of the sample (it is assumed that $\mu_M = \mu_A$),

m_{hkl} and m_{HKL} are the multiplicity factors,

v_M and v_A are the cell volumes,

d_M and d_A are the d spacings,

F_M and F_A are the structure factors for the hkl and HKL lines respectively,

B_M and B_A are the Debye-Waller factors,

$LP(\theta_M)$ and $LP(\theta_A)$ are the Lorentz-Polarization factors which, for the diffractometer used in this work, are given by $LP(\theta) = (1 + \cos^2 2\theta \cos^2 2\theta_{mono}) / \sin 2\theta \cos \theta$ where θ_{mono} is the Bragg angle of the graphite diffracted beam monochromator (i.e. $\theta_{mono} = 13.3^\circ$).

To simplify the analysis, the equations [4.1] may be abbreviated to

$$I_M^{hkl} = C m_{hkl} V_M K_M^{hkl} \text{ and } I_A^{HKL} = C m_{HKL} V_A K_A^{HKL} \quad [4.2]$$

where K_M^{hkl} and K_A^{HKL} contain terms such as the structure factor and cell volume that can be calculated from the known structures of the two phases. In principle, therefore, by measuring the integrated intensities of any two lines in orthodontic steel, the volume fraction (or mass fraction) of either the martensite or austenite phases can be determined from

$$\frac{I_M^{hkl}}{I_A^{HKL}} = \frac{V_M}{(1 - V_M)} \frac{K_M^{hkl}}{K_A^{hkl}} \quad [4.3]$$

The values of these parameters are discussed in Appendix 4.1

4.2.2. Results and analysis.

The integrated intensity for the austenitic and martensite phases (I_M^{hkl} and I_A^{HKL}) in WS, WF, GS and GF wires are obtained from the XRD spectra (figs. 4.3, 4.4, 4.5 & 4.6) by using the X-fit program as following tables 4.1:

Table 4.1:

WS									
	Austenitic phase					Martensitic phase			
2θ(°)	43.7	50.8	74.7	90.6	95.9	44.6	64.8	82	
I(Area)	5950	819	299	263	614	1048	90	89	
WF									
	Austenitic phase					Martensitic phase			
2θ(°)	43.8	50.9	73.6	90.6	96.1	44.9	64.5	82.5	99.6
I(Area)	1324	134	135	51	125	2502	88	88	287
GS									
	Austenitic phase			Martensitic phase					
2θ(°)	43.7	50.9	74.7	44.6					
I(Area)	1464	258	193	95					
GF									
	Austenitic phase					Martensitic phase			
2θ(°)	43.7	50.9	74.9	90.4	96.1	44.7			
I(Area)	1901	364	57	294	185	503			

The K value is a constant for each line (X) of each phase.

$$K = \frac{[Cm_{hkl}LP(\theta_M)F^2e^{-B/2d^2}]}{v_M^2}$$

K value are calculated as in the following table 4.2:

Table 4.2:

Austenitic phase					
hkl	111	200	220	311	222
2θ	43.68	50.78	74.7	90.62	95.96
v _A	46.66	46.66	46.66	46.66	46.66
m _A	8	6	12	24	8
LP _A	11.04	7.945	3.608	2.814	2.73
f	16.45	15.37	12.53	11.46	11.07
F ² _{XA}	16f ² 4329.64	16f ² 3779.8	16f ² 2512.01	16f ² 2101.31	16f ² 1960.72
e ^{-2M}	0.96	0.947	0.896	0.86	0.848
K	168.6C	78.37C	44.76C	56.1C	16.68C

Martensitic phase				
hkl	110	200	211	220
2θ	44.9	64.45	82.49	99.56
v _M	23.55	23.55	23.55	23.55
m _{hkl}	12	6	24	12
LP _M	10.55	4.732	3.125	2.71
f	16.16	13.41	11.85	11.4
F _{XM}	4f ²	4f ²	4f ²	4f ²
	1044.6	719.3	561.7	519.84
e ^{-2M}	0.96	0.92	0.88	0.84
K	228.9C	33.81C	66.85C	24.17C

Volume of each phase is: $V_M = \frac{I_{XM}}{K_M}$. The I_{MX} and K_M for each line of each phase are calculated shown in above table 4.1&4.2, The volume of each phase for each line are calculated in the following tables 4.3.

Table 4.3:

WS									
	Austenitic phase					Martensitic phase			
2θ	43.7	50.8	74.7	90.6	95.9	44.6	64.8	82.2	
hkl	111	200	220	311	222	110	200	211	
V _A	35.3/C	10.5/C	6.7/C	4.7/C	36.8/C	4.58/C	2.66/C	1.34/C	
WF									
	Austenitic phase					Martensitic phase			
2θ	43.8	50.9	73.6	90.6	96.1	44.9	64.5	82.5	99.6
hkl	111	200	220	311	222	110	200	211	220
V _A	7.85/C	1.7/C	3.03/C	0.9/C	7.5/C	10.9/C	2.62/C	1.32/C	11.8/C
GS									
	Austenitic phase			Martensitic phase					
2θ	43.7	50.9	73.7	44.9					
hkl	111	200	220	110					
V _A	8.68/C	3.29/C	4.32/C	0.416/C					
GF									
	Austenitic phase					Martensitic phase			
2θ	43.8	50.9	73.7	90.6	96.1	44.9			
hkl	111	200	220	311	222	110			
V _A	11.3/C	4.65/C	1.27/C	5.25/C	11.1/C	2.19/C			

It is clear from table 4.3 that the value of the volume determined for one phase is different from the separate peaks of the same wire. This implies that

the hypothesis of the XRD pattern as a powder pattern is not correct. This tells us that specimen still has texture even though an attempt was made to make the specimen closely resemble powder by mounting the same area of the same wires at three different angles; level, upright and at an angle of 45° , and by using the spinning stage.

Because of this difficulty, it is impossible to average these very different values to get an average volume of the austenitic phase. Thus, the volume of martensitic or austenitic phase can not be obtained from the above calculations. However, we can attempt to overcome this problem by comparing the different volume of martensitic phase between starting and final wires.

Figures 4.3 & 4.5 are the XRD spectra from the Wilcock and German starting wires. Figures 4.4 & 4.6 are for the Wilcock and German final wire. These four XRD spectra show that all of the wires are (111) austenite and (110) martensite textured. In the WS and GS wire's spectra, the X ray counts of (111) austenite peak are 583 and 185, and the count of (110) martensite peak are 35 and 7. In the WF and GF wire's spectrum, the count of (111) austenite peak are 48 and 140, and the (110) martensite peak are 180 and 35. The counts of these peaks tell us that the (111) austenite peaks intensity of the final Wilcock and German wires (WF & GF) are always lower than those of the starting wires (WS & GS). At same time, the (110) martensite peaks intensity is always increased in the final wires.

The wire drawing process normally increases the (111) austenite and (110) martensite texture which are parallel, or nearly parallel to the wire axis [4.3, 4.13]. Thus, the (111) austenitic peak intensity decreases in the final wires

rather than increases due to occurrence of the phase transformation from austenite into the martensite during the wire drawing process. This means the final wires should have more volume martensitic phase than their starting wires.

4.3. The asymmetrical diffraction approach

4.3.1. Measurement Theory & Experimental Technique.

In the samples both phases displayed strong textures along the axis of the wires corresponding to austenite [111] and martensite [110]. For very narrow wires, e.g WF wire, the degree of preferred orientation during manufacture is often so strong that only the 111-austenite line and the 110-martensite line can be observed in diffractometer patterns taken of wire cross-sections. In such circumstances, accurate quantitative analysis based on the random crystal model, represented by equation [4.1], cannot be carried out without a large correction for preferred orientation. The 111-austenite line and the 110-martensite lines are the habit plane as the stress induced phase transformation results in the 111 austenite plane converting into a 110 martensite plane. Although there is an orientation change during transformation, the actual orientation distribution of the 111 austenite and 110 martensite planes will be approximately the same although the martensite orientation distribution is expected to be a few degrees broader. For accurate quantitative analysis however, allowance should be made for any differences in the orientation distribution of the two phases. This is the approach adopted in the present investigation.

The four wires used were the same as in the previous experiment: WS(1.62mm), WF(0.52mm), GS(2.18mm) and GF(0.88mm). All the XRD

data were collected by reflection diffraction using a conventional Siemens D 5000 diffractometer with a graphite diffracted beam monochromator. Two forms of sample were prepared by embedding wires in an epoxy resin composite and then chemically polishing the exposed wires to obtain a smooth undamaged surface. Illustrations of the two types of sample are shown in Fig. 4.7. After a preliminary investigation of the XRD patterns and the results from each type of specimen, it was decided to use only cross-section specimens shown in Fig. 4.7a. The reason is in specimens of type (a) the austenite 111 line is much more intense than the same line in type (b), whereas the martensite 110 line is approximately the same intensity in both specimen types. This arises because the normals to the 111 austenite and 110 martensite planes are strongly orientated along the axes of the wires. Given this condition then other members of the austenite $\{111\}$ family will be centred about angles of 70.5° , 109.5° and 180° with respect to the axis of the wire. There are fewer crystals therefore oriented with any of the $\{111\}$ family at 90° to the wire axis. Alternatively, for every 110 plane oriented along the wire axis there will be other members of the $\{110\}$ family at 90° and 180° . Hence the 110-martensite line tends to be equally intense in both longitudinal and cross-section specimens. On the whole, cross-sectional specimens of type (a) give the strongest combination of 111 austenite and 110 martensite lines and are easier to interpret because of the well-defined and reproducible nature of the preferred orientation. As the intensity of lines other than the 110 martensite and the 111 austenite can be very small, all the quantitative analysis has been done using the integrated intensities of these two lines only. Fortunately these lines can be measured with good precision which is particularly important for measuring the preferred orientation

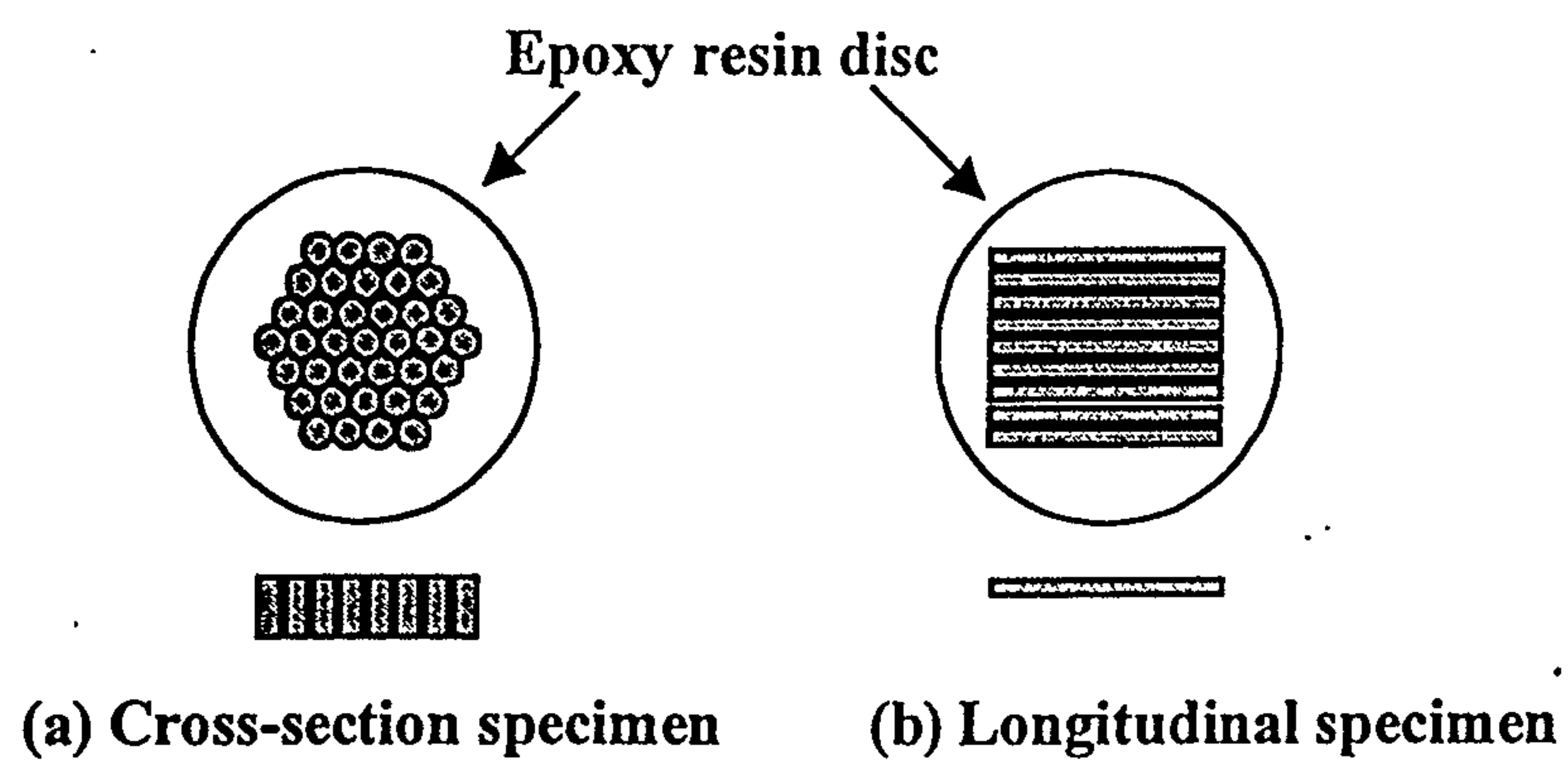


Fig.4.7 Types of specimens prepared for XRD analysis in experiment 2 to measure the martensite/austenite content in orthodontic stainless steels.

distribution where the diffractometer is set-up in an asymmetric condition thereby causing defocussing and diminished intensities.

The procedure developed here for correcting the observed integrated intensities for preferred orientation depends on being able to determine the preferred orientation distributions of the austenite and martensite phases in the wires. A unique aspect of the present procedure is the development of a correction mechanism that compensates not only for the preferred orientation, but also for the finite size and slightly irregular area shape of the specimens.

As the specimens are always set spinning about an axis perpendicular to the specimen surface, the preferred orientation distribution of each phase will depend only on the angle ψ between the normal to the specimen (i.e. the wire axis) and the normals to the hkl planes. The orientation distribution can be derived by carrying out asymmetric diffraction so that crystals with their planes oriented at an angle ψ to the surface normal come into the diffraction condition [4.6,4.7,4.8]. In the present samples asymmetric diffraction has been used to determine the integrated intensities, $I_M^{110}(\psi)$ and $I_A^{111}(\psi)$, of the 110 martensite and 111 austenite lines as function of the off-set angle ψ between the normal to the specimen and the diffraction vector S as shown in Fig.4.8.

Under the asymmetric diffraction conditions shown in Fig.4.8, diffraction profiles from a conventional diffractometer are defocussed and broaden with increasing ψ , particularly at low 2θ angles. An example of the 110 martensite and 111 austenite diffraction profiles obtained from WS and WF orthodontic wires for a range of off-set angles ψ from 0° to 12° is shown in

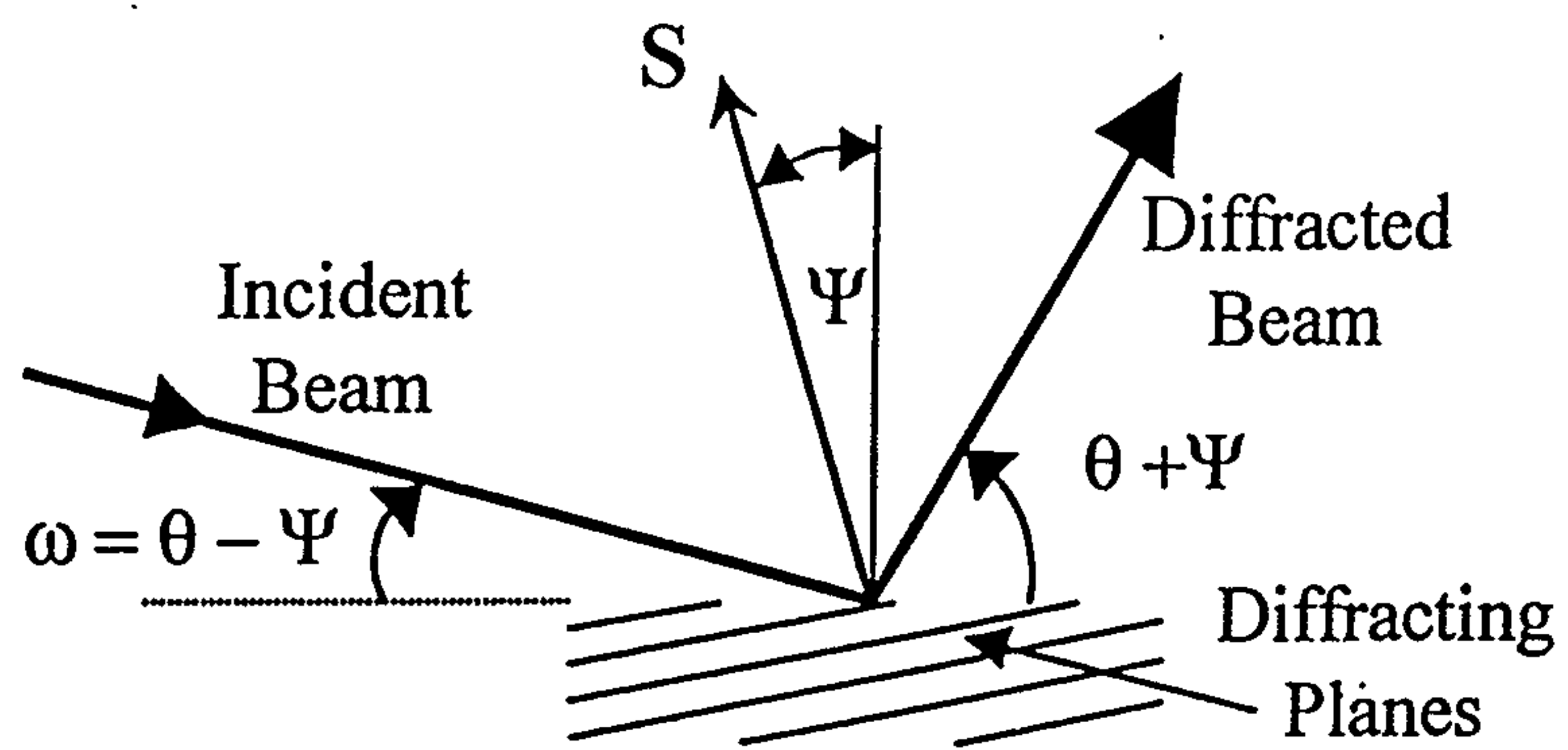


Fig. 4.8 Asymmetric diffraction to measure the intensity diffracted from hkl planes at an angle ψ to the surface of the specimen.

Fig.4.9. Owing to the high concentration of dislocations in the wires, the diffraction lines were very broad (FWHM $\approx 0.5^\circ$ or more) and the defocussing and instrumental broadening was overshadowed by the specimen diffraction broadening. All the measured X-ray profiles $D(2\theta)$ were symmetric and could be accurately fitted by representing each emission line in the $\text{CuK}\alpha$ spectrum with a pseudo-Voigt function [4.9],

$$\text{i.e. } D(2\theta) = \gamma L(2\theta) + (1-\gamma)G(2\theta) \quad [4.4]$$

where $L(2\theta)$ = Lorentzian function and $G(2\theta)$ = Gaussian function. All the integrated intensities, $I_M^{110}(\psi)$ and $I_A^{111}(\psi)$, were therefore determined from profile fitting using the X-ray software package XFIT [4.10, 4.11].

In specimens with randomly oriented crystallites the integrated intensity $I_X(\psi)$ of the line from phase X (i.e. $X = A$ or M) varies with the off-set angle ψ as: [10]

$$I_X(\psi) = I_X^{hkl} (1 + \tan \psi \cot \theta) \quad [4.5]$$

where I_X^{hkl} is the integrated intensity obtained under symmetric diffraction conditions at $\psi = 0^\circ$ for the hkl line in phase X for random crystals. This variation with ψ arises from changes in X-ray absorption and beam spread W across the sample surface when the X-ray beam is incident at an angle ω which is different from the Bragg angle θ (see Fig. 4.10). In the present samples the crystallites in the wires are not randomly oriented. Also, the specimens have a finite lateral size so that the incident beam can extend beyond the specimen, as illustrated in Fig. 4.10, giving a reduced integrated

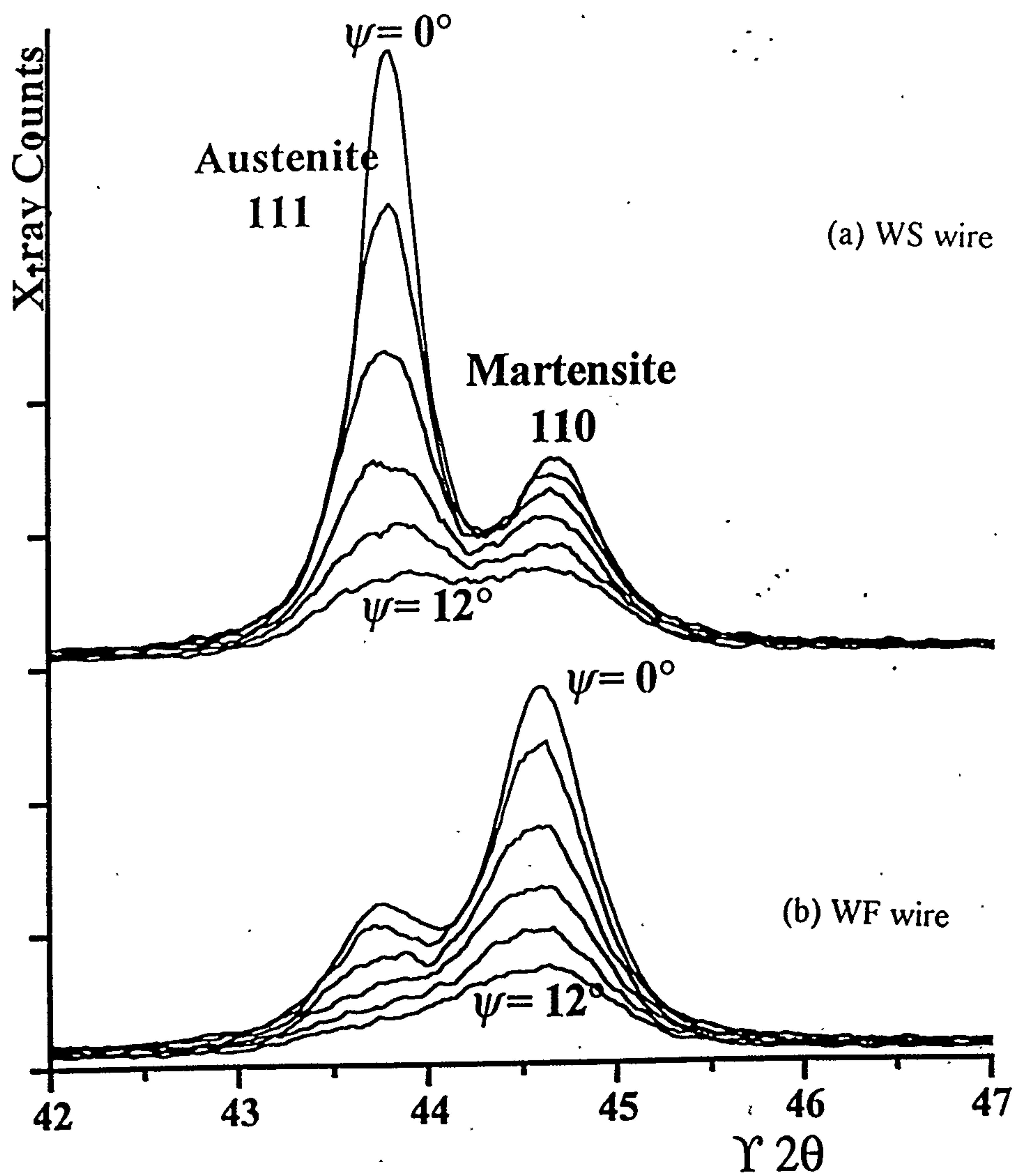


Fig. 4.9 The 110 martensite and 111 austenite lines from an orthodontic wire recorded at different offset angles ψ between 0° and 12° at steps of 2° . The plot for $\psi = 2^\circ$ is omitted for clarity as the profile is almost the same as for $\psi = 0^\circ$.

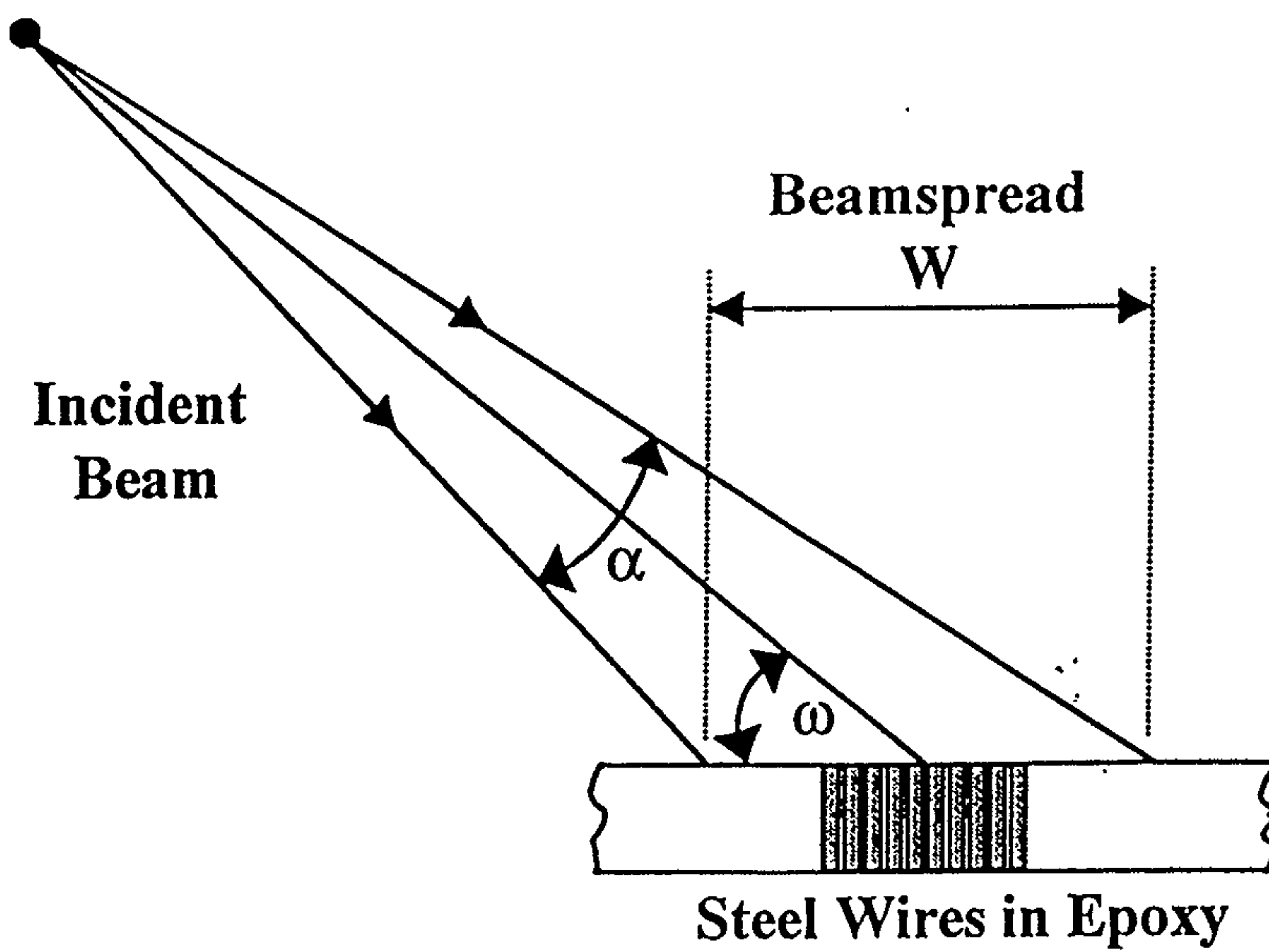


Fig. 4.10 Spread W of the incident beam across a specimen surface for a diffractometer, of radius R , set at an equatorial divergence α and an incident angle ω .

intensity relative to an “infinite” specimen. Under these circumstances equation [4.5] for phase X has to be modified to,

$$I_X(\psi) = f(W)P_X(\psi)I_X^{hkl}(1 + \tan \psi \cot \theta) \quad [4.6]$$

where $f(W)$ represents the fraction of the beam's footprint incident on the specimen when the beams spread is W ; and $P_X(\psi)$ is the ratio of the number of crystallites oriented in the direction of the diffraction vector S (fig.4.8) relative to the random crystal model.

To apply equation [4.6], it is necessary to determine $f(W)$ for each specimen. The effect on the integrated intensity of the incident beam spreading across and beyond the limits of the sample can be characterised by measuring the integrated intensity $I(\alpha)$ of a profile as a function of the angle of divergence α . At any divergence angle α , the beamwidth W is given by the relation,

$$W = \frac{\alpha R \operatorname{cosec} \omega}{1 - (\alpha/2)^2 \cot^2 \omega} \quad [4.6]$$

where ω is the angle of incidence and R is the radius of the diffractometer. At small α , when the beam is contained within the specimen, the integrated intensity $I(\alpha)$ increases linearly with α . For an infinite specimen $I(\alpha)$ would continue to increase linearly with α and it is this line that defines $f(W) = 1$. For a finite specimen the rate of increase of $I(\alpha)$ with α will decrease and then flatten off as the beam spreads beyond the limits of the diffracting sample. Provided the region over which $f(W) = 1$ is sufficiently long to clearly identify the linear region in $I(\alpha)$ then, by extrapolating the linear portion of $I(\alpha)$ to large α , to identify $I(\alpha)_{\text{extrap}}$, the correction factor $f(W)$ is given by,

$$f(W) = \frac{I(\alpha)_{\text{measured}}}{I(\alpha)_{\text{extrap}}} \quad [4.7]$$

In the present work, $f(W)$ was measured separately for each of the four wire specimens using a Siemens D5000 X-ray diffractometer operating in a symmetric mode (i.e. $\omega = \theta$) with the sample spinning and fitted with stepper motor controlled divergence slits which can be automatically incremented in steps of divergence angle (α) 0.1° from 0.2° up to 3° . Thus, in this experiment, the intensity will increase with divergence angle α increase and the specimen will stay in the symmetric mode. Either the 111 austenite or the 110 martensite profile was measured at each α to determine $I(\alpha)$ depending which line was the stronger. The results obtained are given in Fig.4.11a but in terms of the integrated intensity $I(W)$ with beamwidth W , and the expected variation $I(W)_{\text{extrap}}$ for an infinite specimen. Fig. 4.11b. shows the form of $f(W)$, determined from $I(W)$ and $I(W)_{\text{extrap}}$, which for convenience was fitted with a sixth order polynomial. During the determination of $f(W)$ the divergence angle α is a variable term and ω is fixed at θ . When the diffractometer is set up to measure the integrated intensity $I_X(\psi)$ under asymmetric conditions, the angle of divergence is fixed at α_0 and W varies because of the changing angle ψ . The appropriate $f(W)$ term correspond to the value of W given by equation [4.6] with $\alpha = \alpha_0$ (typically 1°) and $\omega = (\theta - \psi)$.

The principal objective of this work is to accurately determine the ratio of the integrated intensities, I_A^{111} / I_M^{110} , of the 111 and 110 lines of the austenite and martensite phases that would be obtained if there were no preferred orientation. From the asymmetric diffraction measurements of $I_X(\psi)$ and

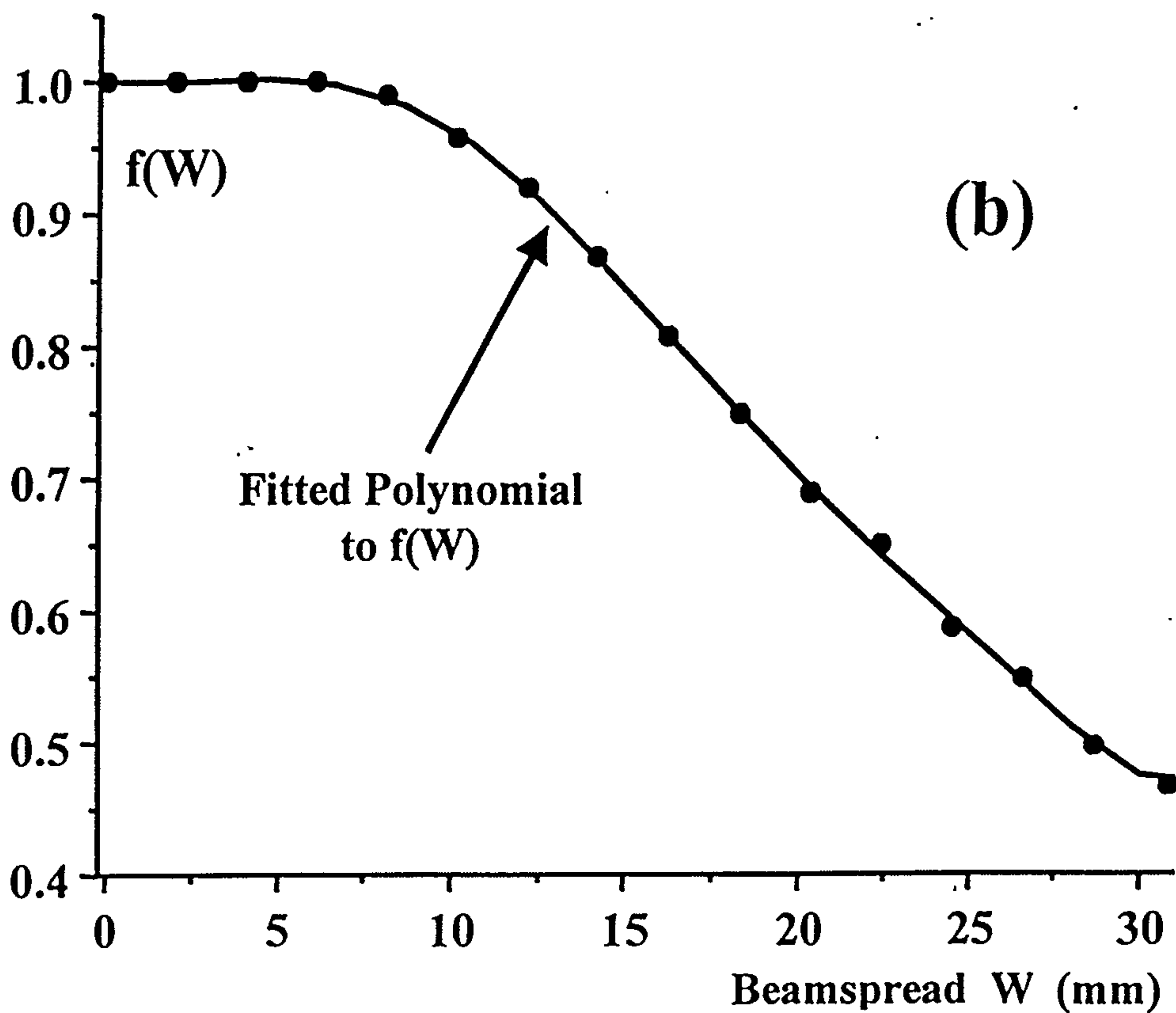
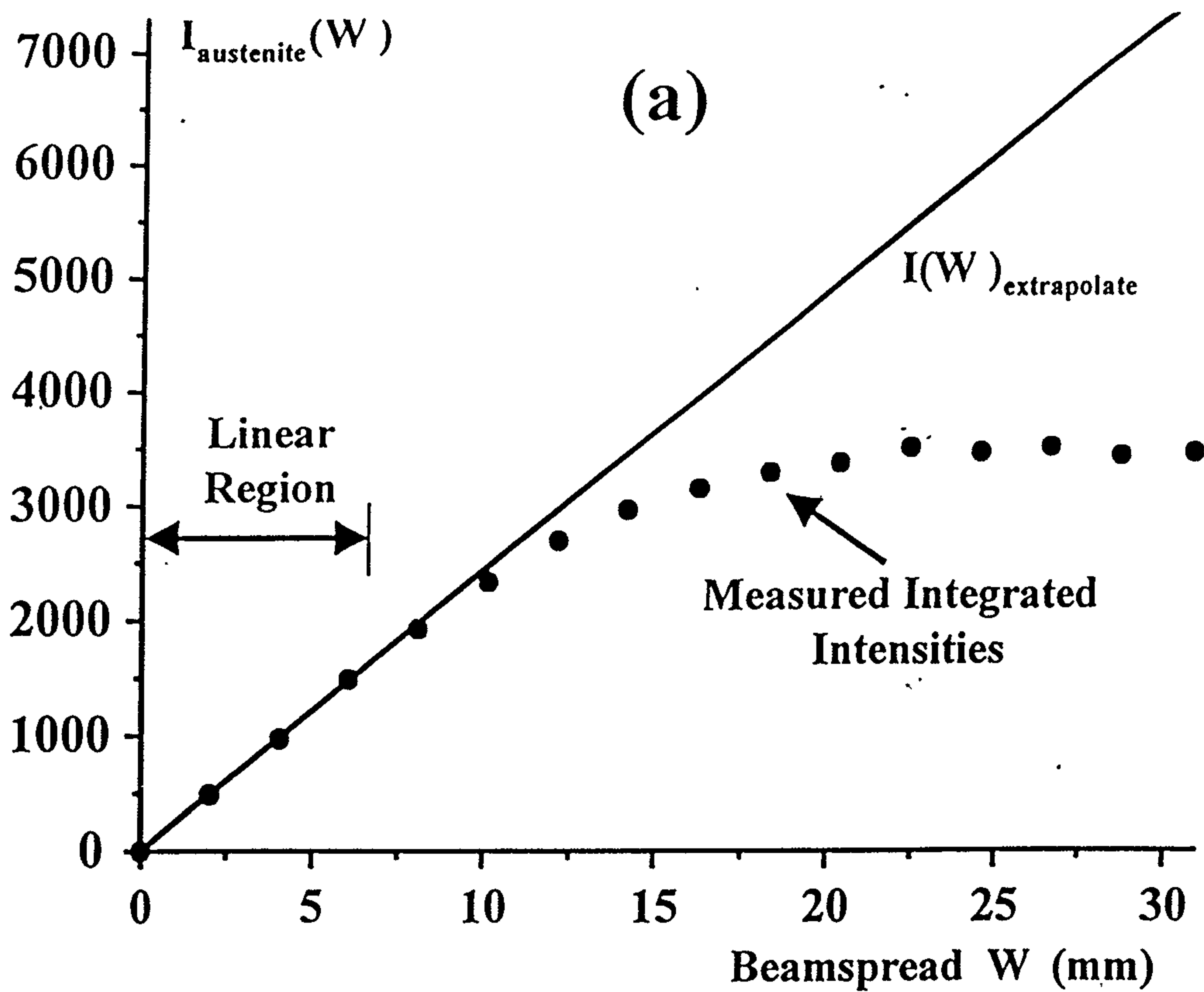


Fig. 4.11 (a) Integrated intensity $I(W)$ measured at different levels of the beamspread W of the incident beam on one of the orthodontic wire samples. The beamspread was varied by increasing the divergence angle α of the beam. (b) Measured correction factor $f(W)$ for finite specimen size when the spread of the incident beam is W .

$f(W)$ for each phase X, it is possible to determine the texture function $\Gamma_X(\psi) = P_X(\psi) I_X^{hkl}$ for each phase over a range of ψ angles using [4.6],

$$\Gamma_X(\psi) = P_X(\psi) I_X^{hkl} = \frac{I_X(\psi)}{f(W_X)(1 + \tan \psi \cot \theta_X)} \quad [4.8]$$

In specimens with random crystallites the orientation distribution term $P_X(\psi) = 1$ at all ψ so that $\Gamma_X(\psi) = I_X^{hkl}$. In addition, all planes in the $\{hkl\}$ family contribute to the intensity term $\Gamma_X(\psi)$. When the crystallites are very strongly oriented the integrated intensity $\Gamma_X(\psi)$ at $\psi = 0^\circ$ is enhanced and $P_X(\psi) \gg 1$ at $\psi = 0^\circ$. However, the orientation function $P_X(\psi)$ decreases to zero very rapidly within a few degrees.

To convert the results for a strongly oriented crystal to the equivalent random integrated intensity I_X^{hkl} / m_{hkl} , it is necessary to determine the average of $\Gamma_X(\psi)$ over all directions in space [4.6]. Unlike the random crystallite case, only one member of the $\{hkl\}$ family is contributing to the diffraction at $\psi = 0^\circ$ in a strongly oriented material and the result of averaging is I_X^{hkl} / m_{hkl} . As all the diffraction measurements are carried out with the specimen spinning in its own plane, the measured $\Gamma_X(\psi)$ values already represent an average value at a fixed ψ over the longitudinal coordinate ϕ around the polar axis $\psi = 0$. The mean value of $\Gamma_X(\psi)$ over the surface of a sphere is therefore defined by the equation,

$$\frac{1}{4\pi} \int_0^\pi 2\pi \sin \psi \Gamma_X(\psi) d\psi = \frac{I_X^{hkl}}{m_{hkl}} \quad [4.9]$$

This above equation is derived from the following:

From the fig.4a, we observed that $dA = x d\psi 2\pi X \sin\psi$, where $0 \leq \psi \leq \pi$.

It follows that $\Gamma(\psi)dA = 2\pi x^2 \Gamma(\psi) \sin\psi d\psi$

By integration we can find the total intensity over the sphere as $\int_0^\pi \Gamma(\psi) dA$

So, the average intensity = Total intensity / Area of sphere = $\int_0^\pi \Gamma(\psi) dA / 4\pi x^2$

$$\begin{aligned}
 &= \frac{\int_0^\pi \Gamma(\psi) dA}{4\pi x^2} \\
 &= \frac{2\pi x^2 \int_0^\pi \Gamma(\psi) \sin(\psi) d(\psi)}{4\pi x^2} \\
 &= \frac{1}{4\pi} \int_0^\pi 2\pi \sin \psi \Gamma_x(\psi) d\psi \\
 &= \frac{I_x^{hkl}}{m_{hkl}}
 \end{aligned}$$

In the orthodontic wires investigated here the texture is not always strong enough for the function $\Gamma_x(\psi)$ to decay rapidly to zero and other planes in the $\{hkl\}$ family contribute a small amount to the integrated intensity over the range of measurement in ψ . In this case the observed function $\Gamma_x(\psi)$ is a sum of Γ functions each representing a different sub-group of hkl planes in the $\{hkl\}$ family. For the austenite 111 line between $\psi = 0^\circ$ and 12° , $\Gamma_A(\psi)$ is represented as the sum of two functions,

$$\text{i.e.} \quad \Gamma_A(\psi) = \Gamma_{A,0}(\psi) + \Gamma_{A,70.5}(\psi) \quad [4.10a]$$

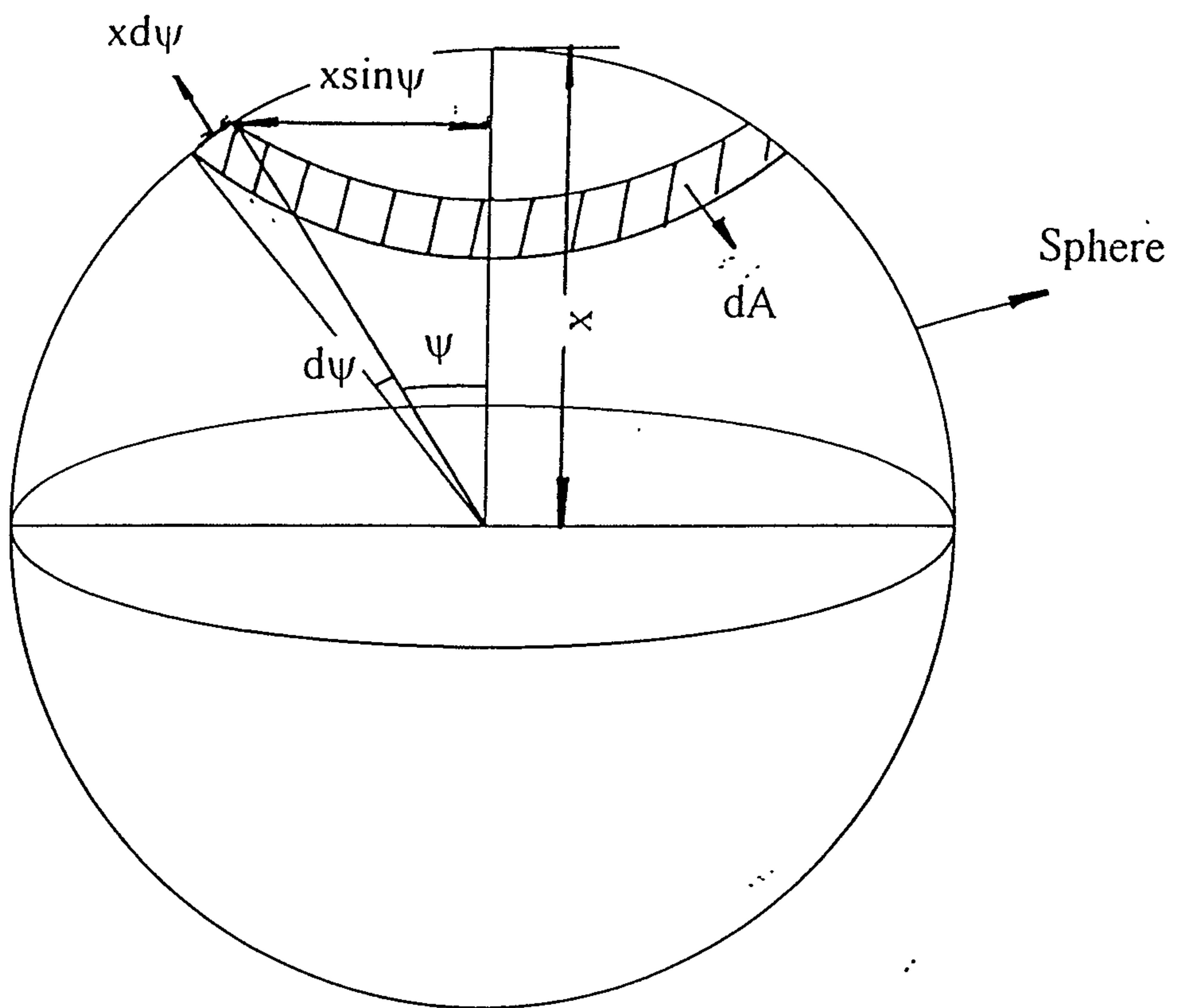


Fig. 4a Schematic drawing for derive the equation of [4.9]

where $\Gamma_{A,0}(\psi)$ represents the distribution of the (111) planes centred about $\psi = 0^\circ$, and $\Gamma_{A,70.5}(\psi)$ represents the distribution of the $(11\bar{1})$, $(1\bar{1}1)$ and $(\bar{1}11)$ planes centred about $\psi = 70.5^\circ$. Although there are also distributions about $\psi = 109.5^\circ$ and 180° representing other members of the $\{111\}$ family, these can be neglected in the present case as their contributions at $\psi \leq 12^\circ$ are very small. An illustration of $\Gamma_A(\psi)$ and the contributions of the component functions $\Gamma_{A,0}(\psi)$ and $\Gamma_{A,70.5}(\psi)$ corresponding to the present samples is given in Fig. 4.12. The two sets of broken lines are obtained by using the X-fit program, they represent the exactly peaks at 0° and 70.5° . Similarly, for the martensite 110 line, $\Gamma_M(\psi)$ is represented as the sum,

$$\text{i.e.} \quad \Gamma_M(\psi) = \Gamma_{M,0}(\psi) + \Gamma_{M,60}(\psi) \quad [4.10b]$$

where $\Gamma_{M,0}(\psi)$ represents the distribution of (110) planes and $\Gamma_{M,60}(\psi)$ represents the distribution of the (101), (011), $(01\bar{1})$ and $(10\bar{1})$ planes.

Under these circumstances, the appropriate function for calculating I_X^{hkl} / m_{hkl} through equation [4.9] is $\Gamma_{X,0}(\psi)$ rather than $\Gamma_M(\psi)$. The question therefore is, how can $\Gamma_{X,0}(\psi)$ be extracted from the experimental results $\Gamma_X(\psi)$? In the present results, the dominant contributions to $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$ are $\Gamma_{M,0}(\psi)$ and $\Gamma_{A,0}(\psi)$, respectively, and the higher terms, $\Gamma_{M,60}(\psi)$ and $\Gamma_{A,70.5}(\psi)$, behave as a “background contribution” which typically contribute $<2\%$ of the measured $\Gamma_M(\psi)$ or $\Gamma_A(\psi)$ values (see Fig. 4.12). As a first approximation therefore, it can be assumed that over the fitted range $0^\circ \leq \psi \leq 12^\circ$,

$$\Gamma_M(\psi) \approx \Gamma_{M,0}(\psi) \quad \text{and} \quad \Gamma_A(\psi) \approx \Gamma_{A,0}(\psi) \quad [4.11]$$

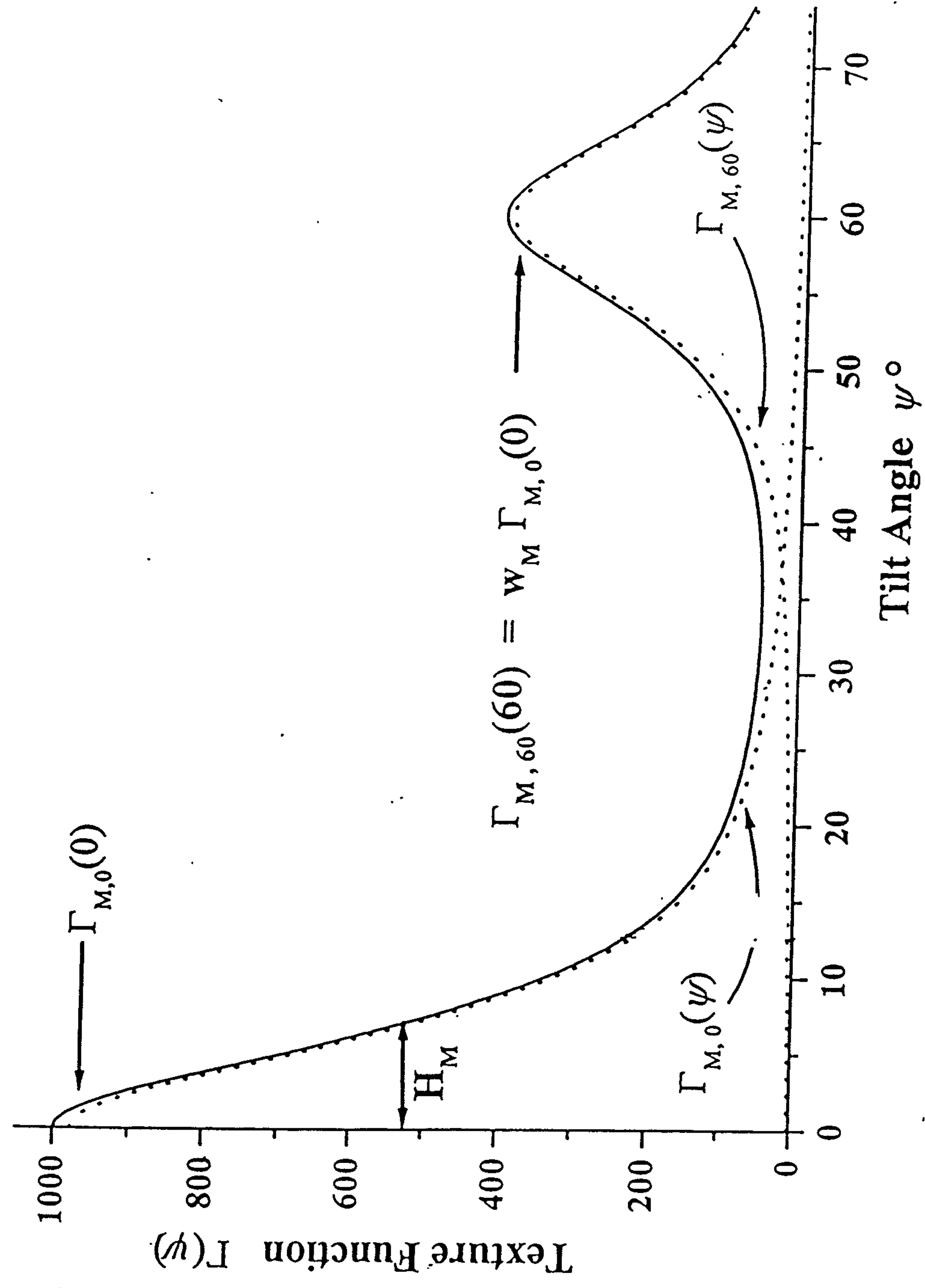


Fig. 4.12 An illustration of the function $\Gamma_M(\psi)$ for a martensite phase showing it as a sum of the texture function $\Gamma_{M,0}(\psi)$ for the 110 planes, and $\Gamma_{M,60}(\psi)$ for the distribution of the (101) , (011) , $(01\bar{1})$ and $(10\bar{1})$ planes centred on $\psi = 60^\circ$.

In the examples discussed in this paper the error introduced into the measured volume fraction of martensite V_M by neglecting the higher order terms in $\Gamma_X(\psi)$ is no more $\sim 2\%$. Further discussion on the magnitude of this systematic error is given later.

In practice the procedure adopted to determine V_M is to first fit the texture functions $\Gamma_A(\psi)$ and $\Gamma_M(\psi)$ over the measured range in ψ . Pseudo-Voigt functions are used here as these fit well with a shape factor γ between 67% and 100% Lorentzian [4.9]. The fitted functions are then calculated at 0.1° steps over the range $\psi = 0$ to 180° , weighted by $\sin\psi$, before calculating the numerical integral Ω_X ($X = M$ or A) given by,

$$\Omega_X = \int_0^{180} \sin\psi \Gamma_X(\psi) d\psi \quad [4.12]$$

The full expression used to determine the volume fraction V_M of martensite when equation [4.3] is expressed in terms of measured and calculated parameters becomes,

$$\frac{\Omega_M}{\Omega_A} = \frac{V_M}{1-V_M} \frac{K_M^{hkl}}{K_A^{HKL}} \quad [4.13]$$

The fitting and numerical integration associated with the above analysis is usually done using the numerical analysis and graphics software package ORIGIN 5.0.

In the present samples the preferred orientation is relatively strong and the texture widths H_A and H_M , as defined in Fig.4.12, are all within the band 5° to 13° . For this degree of preferred orientation the overlap between the

component functions $\Gamma_{M,0}(\psi)$ and $\Gamma_{M,60}(\psi)$, and between $\Gamma_{A,0}(\psi)$ and $\Gamma_{A,70.5}(\psi)$, is small but not negligible. The basis of the iterative correction procedure developed for compensating for the overlap assumes that the two component functions within any measured $\Gamma_X(\psi)$ have essentially the same shape, but different total areas, so that

$$\Gamma_{A,70.5}(\psi) = \omega_A \Gamma_{A,0}(\psi - 70.5) \quad \text{and} \quad \Gamma_{M,60}(\psi) = \omega_M \Gamma_{M,0}(\psi - 60) \quad [4.14]$$

where w_A and w_M are the relative weights of the component peaks (see Fig.4.12). This assumption is essentially stating that if the distribution of the (110) planes in the martensite phase around $\psi = 0^\circ$ is $\pm 10^\circ$, then the distribution of the (101), (011), $(01\bar{1})$ and $(10\bar{1})$ planes about $\psi = 60^\circ$ will also be $\approx \pm 10^\circ$. On this basis the measured $\Gamma_X(\psi)$ terms can be expressed as,

$$\Gamma_A(\psi) = \Gamma_{A,0}(\psi) + \omega_A \Gamma_{A,0}(\psi - 70.5) \quad \text{and} \quad \Gamma_M(\psi) = \Gamma_{M,0}(\psi) + \omega_M \Gamma_{M,0}(\psi - 60) \quad [4.15]$$

To develop an overlap correction it is necessary to know the relative weights w_A and w_M . These can be estimated on the basis that three planes contribute to $w_A \Gamma_{A,0}(\psi - 70.5)$ so that when averaged over the surface of a sphere this function will be three times that of the average value of $\Gamma_{A,0}(\psi)$. For the equivalent martensite functions, the equivalent ratio is four to one. Under the conditions appropriate for the present sample, the weighting term w_X ($X = A$ or M) can be reduced from the expression,

$$w_X \cong \frac{M_X}{2 \sin \psi_0} \frac{\int_0^{180} \Gamma_{X,0}(\psi) \sin \psi \, d\psi}{\int_0^{180} \Gamma_{X,0}(\psi) \cos \psi \, d\psi} \quad [4.16]$$

where $M_X = 3$ or 4 and, $\psi_0 = 70.5^\circ$ or 60° for austenite and martensite, respectively. In the execution of this correction procedure w_X was first estimated by letting $\Gamma_{X,0}(\psi) = \text{fitted function } \Gamma_X(\psi)$ in equation [4.16]. The measured function $\Gamma_X(\psi)$ was then fitted using the model represented by equation [4.15] to obtain a new fitted form for $\Gamma_{X,0}(\psi)$. An updated value for w_X can then be obtained using equation [4.16] and further iterations carried out to converge on a more accurate form for $\Gamma_X(\psi)$. This was unnecessary for the present samples as one iteration was sufficient to obtain a sufficiently accurate correction.

4.3.2. Results and Analysis

The texture functions $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$ obtained for the four different wires over the range $\psi = 0^\circ$ to 12° are shown in Fig. 4.13. This figure also shows the fitted curves $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$ from which the terms Ω_A and Ω_M were determined. The results of the fitting are given in Table 4.4 along with the calculated volume fraction V_M of martensite in each wire and the breadths, H_M and H_A , of the functions $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$. A number of different results for V_M are presented, which are discussed in more detail later, comparing the present method with the results obtained if no preferred orientation correction is applied. The terms H_M and H_A reflect the degree of texture in the particular phase and represent the values of ψ at which the fitted texture function $\Gamma_{X,0}(\psi)$ decreases to 50% of its initial value at $\psi = 0^\circ$. All the uncertainties quoted in Table 4.4 reflect the precision of the fitting procedure. The atomic scattering factors and associated dispersion corrections used to determine the structure factors for calculating V_M from

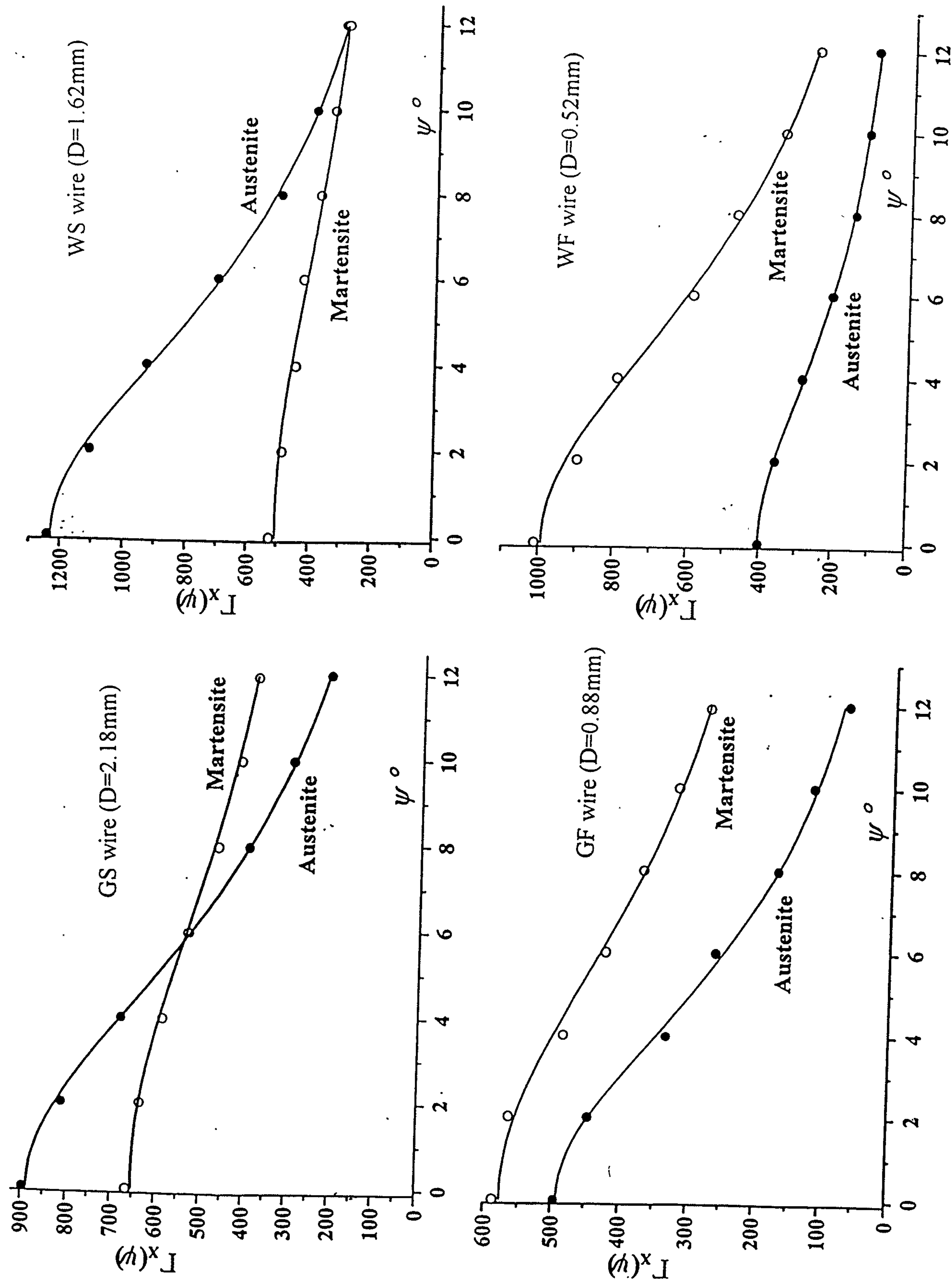


Fig. 4.13 The measured and fitted texture functions, $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$, for each of the four orthodontic wires.

equation [4.13] were obtained from the International Tables for Crystallography Volume C [4.12]. In the calculation of the structure factors it was assumed that the alloying elements in the wires were substitutionally disordered so that,

$$F_{\text{austenite}} = 4f \exp(-B_A \sin^2 \theta / \lambda^2) \text{ and } F_{\text{martensite}} = 2f \exp(-B_M \sin^2 \theta / \lambda^2)$$

where $f = 0.700f_{\text{Fe}} + 0.181f_{\text{Cr}} + 0.078f_{\text{Ni}} + 0.014f_{\text{Mn}} + 0.027f_{\text{Si}}$. It was assumed that temperature parameters $B_A = B_M$ and that these values will be $\approx 0.35 \text{ \AA}^2$, the same as Fe at room temperature [4.14]. The lattice parameters used for calculating the cell volume, $a_{\text{aust}} = 3.586 \text{ \AA}$ and $a_{\text{mart}} = 2.871 \text{ \AA}$, were measured directly from the XRD pattern. As the 111 austenite and 110 martensite lines $< 1^\circ 2\theta$ apart, any error in the choice of B parameter or the composition will only have a small effect on the volume fraction V_M determined through equation [4.13] as this equation contains the ratio F_M/F_A which ≈ 0.5 irrespective of the temperature parameter B or the alloy composition.

In all the wires the preferred orientation of the austenite phase is stronger than the martensite phase. The width H_A of the austenite phase is 5.7 after extrusion and 6.9 before extrusion, ie. the extrusion process only increases the texture by a relatively small amount. The texture of the martensite phase is less pronounced than the austenite phase and, with the exception of the finer type W wire, $H_M - H_A \geq 5^\circ$. This difference is probably the result of the orientation change that occurs during the austenite-martensite transformation. The actual effect of the extrusion process on the martensite orientation is quite different for the two type wires. In the Type W wire, in particular, the orientation width H_M decreases by almost 6° on extrusion

where as the change in the type G wire is $\sim 1.7^\circ$ and only slightly larger than the change for the austenite phase in this wire.

The fact that the degree of texture in the martensite and austenite is quite different for three of the wires is the main factor that makes correction for preferred orientation very necessary. Column (b) of the results for V_M in Table.4.4, which is corrected for both preferred orientation and overlap, is the most accurate of the three sets. The results for V_M , labelled “random crystals” (i.e. column (c)) with no orientation correction, were obtained by substituting the integrated intensities for martensite and austenite at $\psi = 0$ into the conventional quantitative analysis equation represented by equation [4.3]. When the degree of texture of the two phases is similar, as is almost the case for the finer Type W wire, the volume fraction V_M obtained by assuming no preferred orientation will be similar to the result obtained after correction for preferred orientation. Under these circumstances the preferred orientation correction terms for the austenite and martensite tend to cancel each other. In three of the wires considered here, the texture of the austenite phase is much stronger than martensite, and the preferred orientation correction terms are different for each phase. As a consequence, application of the “random crystal” intensity equation severely under-estimates the martensite content by up to 50%. To within a 10% margin, the volume fraction obtained after correction for preferred orientation, $V_M^{corrected}$, is related to the volume fraction obtained from the “random crystal” intensity equation, V_M^{random} , as,

$$V_M^{corrected} \approx 1.05 \frac{H_M}{H_A} V_M^{random} \quad [4.14]$$

Unfortunately, it was not possible to compare the present results for V_M with those using other techniques. Both magnetic measurements and transmission electron microscopy (TEM) were carried out on the specimens, but neither technique provided reliable results. TEM images were uninformative because of the fine grain structure of the orthodontic wires and the fact that both phases are twin structures of almost identical appearance (ref. Chapter 5). Magnetic measurements on orthodontic wires are difficult to interpret, and measurements carried out on the present wires at another laboratory were clearly inconsistent with the X-ray results. In particular, for one of the specimen shown in Fig. 4.1, magnetic measurements gave zero martensite content which is clearly in error given the magnitude of the martensite peak.

The uncertainties quoted in Table 4.4 for volume fractions V_M of martensite after correction for preferred orientation range from $\pm 4\%$ up to $\pm 8\%$. These uncertainties reflect standard deviations associated with fitting the texture function $\Gamma_X(\psi)$ to determine $\Gamma_{X,0}(\psi)$ and its subsequent extrapolation beyond the measured range to calculate the integral terms $\Omega_X = \int_0^{180} \sin \psi \Gamma_{X,0}(\psi) d\psi$. For the texture widths encountered in the austenite and martensite phases this uncertainty is significantly greater than the correction for overlap between the component functions within each $\Gamma_X(\psi)$ function. To obtain an accurate estimate of the integral Ω_X it is of central importance to be able to predict the shape of the tail of $\Gamma_{X,0}(\psi)$ with reasonable accuracy because of the weighting introduced by the “ $\sin \psi$ ” within the integrand of Ω_X . In the present data, the extrapolated austenite functions $\Gamma_{A,0}(\psi)$ are more reliable

than the corresponding martensite functions $\Gamma_{M,0}(\psi)$. Although both sets of fitted functions extend beyond measurement limit of ψ , the texture widths H_M of all but one of the martensite results are also larger than 12° , the maximum measured ψ . This makes the extrapolation of $\Gamma_{M,0}(\psi)$ beyond 12° very sensitive to the accuracy of both the fitted texture width H_M and the pseudo-Voigt parameter γ_M which describes the degree of Lorentzian character of $\Gamma_{M,0}(\psi)$. At one extreme, when the fitted function is close to Lorentzian (i.e. $\gamma_M \approx 1$) $\Gamma_{M,0}(\psi)$ needs to be extrapolated to $\psi \approx 6H_M$ before decaying to the noise level. Conversely, when the texture function is Gaussian shaped (i.e. $\gamma_M \approx 0$) the function $\Gamma_{M,0}(\psi)$ decays very rapidly within $\psi \approx 3H_M$ and the uncertainty of the extrapolated function is less.

The accuracy of the results presented here, would have been better had CrK α radiation been available ($\lambda = 2.29 \text{ \AA}$). At this wavelength the 111 and 110 lines shift from $2\theta \approx 45^\circ$ up to $\approx 67^\circ$ thereby increasing the upper limit of ψ from 12° up to $\sim 25^\circ$. The only drawback is a larger overlap correction because of the greater contribution of the higher order components $\Gamma_{M,60}(\psi)$ and $\Gamma_{A,70.5}(\psi)$ in the range of measurement. To obtain an accuracy $\approx 5\%$ or less for the volume fraction V_M using the method proposed here, it is recommended that the texture widths, H_M and H_A , be less than 10° and that the measured ψ range be at least twice the largest H_X value. As the texture widths become larger and the measurement range become smaller, the uncertainty in the extrapolated texture functions grows along with the uncertainty in V_M .

Table 4.4 Results obtained after fitting the measured texture function $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$ for each of the four wires. The terms H_M and H_A are a measure of the preferred orientation and represent the ψ angles at which the fitted functions $\Gamma_{M,0}(\psi)/\Gamma_{M,0}(0)$ and $\Gamma_{A,0}(\psi)/\Gamma_M(0) = 0.5$. The three columns labelled V_M are the volume fraction of martensite obtained from the present data, (a) using the present orientation correction assuming $\Gamma_X(\psi) = \Gamma_{X,0}(\psi)$, (b) using the present orientation correction but compensating for overlap between $\Gamma_{A,0}(\psi)$ and $\Gamma_{A,70.5}(\psi)$ and, between $\Gamma_{M,0}(\psi)$ and $\Gamma_{M,60}(\psi)$, (c) using the integrated intensities measured at $\psi = 0^\circ$ and the intensity equation for randomly oriented crystals (equation [3]). The numbers in brackets are the uncertainties that propagate from the uncertainties from fitting the observed texture functions $\Gamma_M(\psi)$ and $\Gamma_A(\psi)$.

Wire Type	H_M°	H_A°	(a) V_M Orientation Correction (No Overlap Correction)	(b) V_M Orientation Correction (+ Overlap Correction)	(c) V_M No Orientation Correction (Random Xtal)
W (1.62 mm)	12.9(3)	6.8(2)	0.58(5)	0.56(5)	0.25(1)
W (0.52 mm)	7.1(2)	6.3(2)	0.75(3)	0.74(3)	0.65(2)
D (2.18 mm)	12.3(3)	6.9(1)	0.72(3)	0.70(3)	0.35(1)
D (0.88 mm)	10.6(2)	5.7(2)	0.83(6)	0.82(6)	0.47(2)

References for chapter 4

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Appendix 4.1.

The volume of the austenitic unit cell is $v_A = a^3 = 3.6^3 = 46.66 \text{ \AA}^3$

The volume of the martensite unit cell is $v_M = a^3 = 2.8664^3 = 23.55 \text{ \AA}^3$
(m_{hkl})

Because both martensite and austenitic phases are cubic structures, the multiplicity factor values are as follows:

hkl	48,	hhl	24,	0kl	24,
0kk	12,	hhh	8,	00l	6.

($LP(\theta_M)$)

The formula for the Lorenz factor is:

$$\frac{1 + \cos^2 2\theta \cos^2 2\theta_m}{\sin^2 \theta \cos \theta}$$

and as in the text, $CuK\alpha$ radiation have been used, ie $2\theta_m = 26.6^\circ$.

(F_M & F_A)

The structure factor for the martensite structure (bcc) is

$$F_{hkl} = \sum_i f_i e^{2\pi i(hx_i + ky_i + lz_i)}$$

$$F = fe^{2\pi i(0)} + fe^{2\pi i(h/2 + k/2 + l/2)}$$

$$= f [1 + e^{\pi i(h+k+l)}]$$

$$F = 2f \quad F^2 = 4f^2 \quad \text{when } h+k+l \text{ is even; and}$$

$$F = 0 \quad F^2 = 0 \quad \text{when } h+k+l \text{ is odd.}$$

The structure factor for austenitic structure (fcc) is

$$F = f [1 + e^{\pi i(h+k)} + e^{\pi i(h+l)} + e^{\pi i(k+l)}]$$

$$F = 4f \quad F^2 = 16f^2 \quad \text{for unmixed indices; and}$$

$$F = 0 \qquad F^2 = 0 \qquad \text{for mixed indices.}$$

The parameter f is the atomic scattering factor. It depends upon the composition of the material and the value

$$\frac{\sin \theta}{\lambda}$$

where in our case $\lambda = 1.54$ (CuK_α radiation).

For 302 stainless steel,

$$f = 0.18f_{Cr} + 0.08f_{Ni} + 0.72f_{Fe} + 0.02f_{Mn}.$$

$$(e^{-2M})$$

The temperature factor is $e^{-2M} = e^{-0.299\sin^2\theta}$ [4.13]

where, θ is Debye characteristic temperature of the substance in degrees Kelvin.

Chapter 5 Transmission electron microscopy investigation of GS, GF, WS and WF wires.

5.1. Introduction

For the analysis of the microstructure of the wires, not only X-ray diffraction (XRD) techniques were used as described in a previous chapter but transmission electron microscopy (TEM) was also applied. TEM techniques can analyse very small microstructural features whereas XRD is more relevant to the analysis of the microstructure of bulk materials.

As mentioned in chapter 4 on XRD analysis, the properties of AISI 302 type stainless steel orthodontic wires are governed by the microstructure of the materials as well as by their chemistry. All the wires are made from the same alloy, AISI 302. This alloy has a standard composition and so the material properties heavily depend on the effect on the microstructure resulting from the manufacturing processes.

In the orthodontic literature, there are very few papers about the TEM investigation of the microstructure of orthodontic wires. This may be due to the difficulty of using TEM for inspecting material with such a small grain size and with the serious deformation microstructure. Moreover, with only about 0.52mm diameter wires, it is very difficult to make a sample for TEM.

But, in the metallurgical literature, 302 austenite stainless steel processed by cold working is a well established material, which has been extensively researched using TEM.[5.1-5.14]. However, in almost all cases, the research

was on material which was less severely deformed than the orthodontic wires which have been deformed down to 0.52mm in diameter.

Type 302 orthodontic wires are produced by a cold drawing process which involves intermediate annealing heat treatments. Under this heavy drawing manufacture process, the microstructure undergoes the following changes:[5.1-5.24]

- a) Texture is introduced.
- b) Twinning and other changes such as the introduction of subgrains may occur to the structure morphology.
- c) The martensite phase(both ϵ and α type martensite phases) forms from the austenite matrix, and other phases such as δ -ferrite (δ -Fe), chromium carbide (Cr_{23}C_6 , Cr_7C_3) and iron carbide (Fe_3C) may appear.
- d) The grain size becomes very small and the shape becomes elongated along the wires axis. The properties of the material become anisotropic as more grain boundaries form in one direction.
- e) A high dislocation density appears.

In this chapter, the microstructure is studied using TEM to determine:

- a). the phases present in the wires,
- b). the size and shape of the grains; and
- c.) the microstructure morphology of the different phases.

The above microstructure information extends down to the atomic level and can be related to the analysis of the differences in mechanical properties of the same material but different diameter wires.

5.2. Materials and Experiment Method

5.2.1. Introduction of transmission electron microscopy (TEM).

The principles of the TEM techniques used during the present work (bright and dark field imaging, selected-area electron diffraction (SAD), and energy-dispersive x-ray spectroscopy (EDS) are introduced in the following sections.

5.2.1.1. Image formation in a transmission electron microscope [5.25-5.29].

In a transmission electron microscope, the almost parallel electron beam is scattered by the specimen. The electrons leaving the specimen are focused by an electromagnetic objective lens to form an image. The image is magnified further by a combined system of intermediate and projector lenses [5.25, 5.26]. Since the intermediate and projector lenses only function as image magnification, one can reduce the real transmission electron microscope system to three essential components for a simplified understanding: incident beam, specimen and objective lens as schematically depicted in fig. 5A. The basis for the production of an image can then be described following the wave-optics theory attributed to Abbe [5.25, 5.26]. For example, all waves leaving the specimen in a given direction are brought together to a point on the back focal plane of the objective lens. Thus in this plane the Fraunhofer diffraction pattern is formed, with maxima where

waves from all parts of the specimen add constructively. Beyond the back focal plane, rays from a common point in the specimen converge on a common point in the image plane. The pattern recorded from the back focal plane is called the electron diffraction pattern and represents a Spherical Edward Sphere through reciprocal space while the pattern in the image plane is in real space (fig. 5A). One can set a circular aperture with a certain desired diameter at the level of the image plane to confine the specimen area from which the diffraction information is obtained. The resulting electron diffraction pattern is then called a selected-area electron diffraction (SAED) pattern and the aperture is called the SAED aperture as shown in fig. 5A.

5.2.1.2. Diffraction contrast: bright-field and dark-field images.

If all the waves leaving the object are recombining again with exactly the same relative amplitudes and phases, the image gives an exact reproduction of the transmission function of the object. However, this is limited in practice mainly by the objective lens and its aberrations. The effect can be characterised by an objective lens transfer function. There are several predominant factors involved in the transfer function limiting the image resolution. These are

- a. the size (diameter) of the objective aperture
- b. the spherical aberration of the objective lens
- c. the chromatic aberration of the objective lens
- d. the incident-beam divergence.

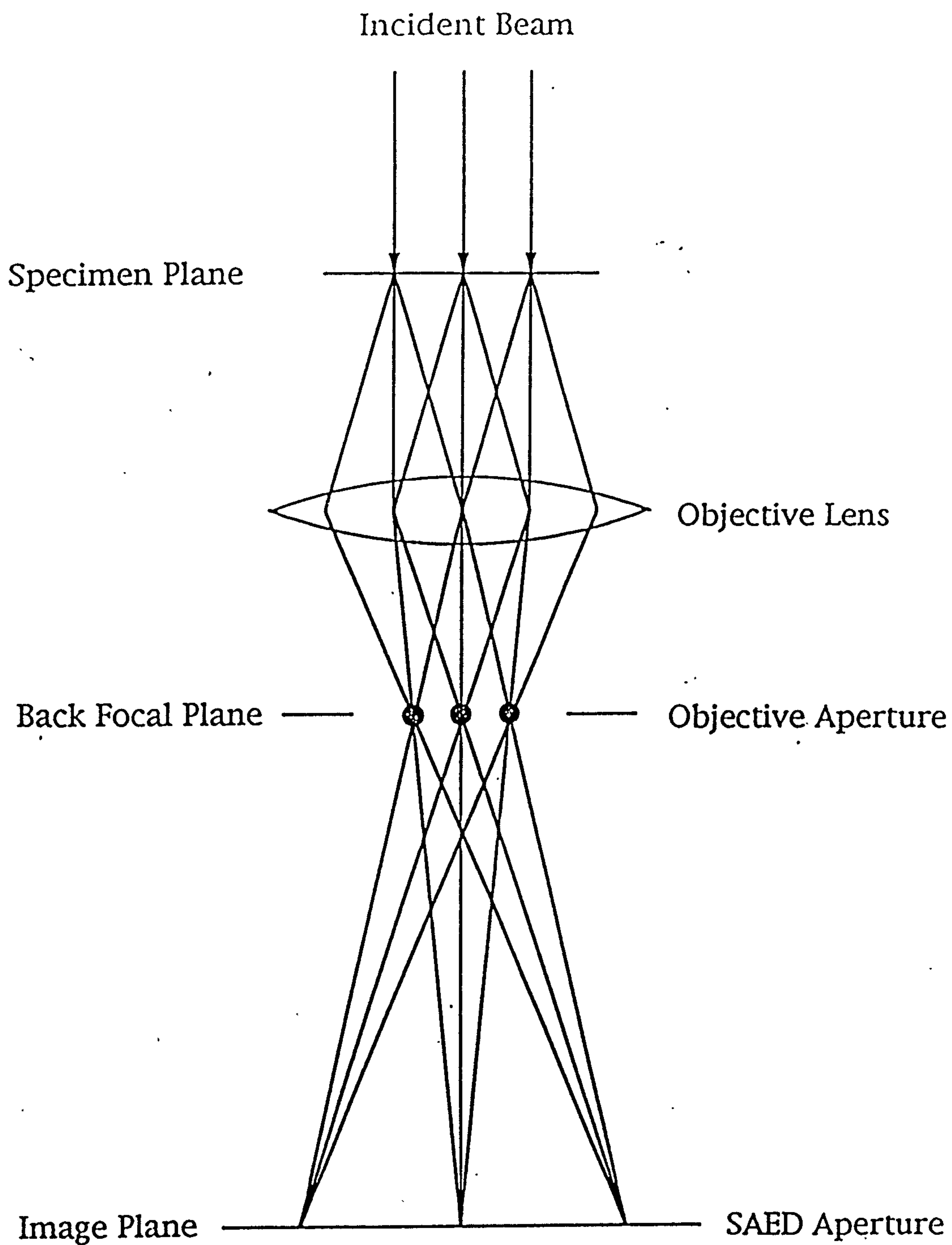


Fig. 5A Ray diagram illustrating the formation of a Fraunhofer diffraction pattern on the back focal plane of a lens and the subsequent formation of an image.

For instance, the finite objective apertures will cut off the waves with high diffraction angles falling beyond the area of the objective aperture. Since some of the scattered beams have been removed and the amplitude of the image signal is reduced, the resulting contrast is called 'amplitude contrast'. An example is 'diffraction contrast', the scheme for which is shown in fig.5B. The inserted objective aperture (from 10-100 μm in a transmission electron microscope) makes a cut-off of the elastic Bragg reflections; therefore the final image is formed only by the direct beam and any low angle inelastic scattering (fig. 5Ba). This type of image is called a *bright field image* [5.25, 5.26].

An image can also be formed by any of the diffracted beams by either displacing the objective aperture to receive the beam, or better by tilting the illumination so that the required beam passes down the axis of the objective lens (fig.5Bb). The resulting image is known as a '*dark-field image*' [5.26]. Dark-field imaging is an essential technique to unravel complex diffraction patterns, e.g. to analyse the origin of additional diffraction spots due to various phases.

5.2.1.3. Selected-area electron diffraction (SAD) [5.25-5.27].

Geometrically, an electron diffraction pattern can be considered as an intersection of the reciprocal lattice of the studied crystal with the Ewald sphere which has a radius $k = 1/\lambda$ (see fig. 5C). In practice, the recorded diffraction pattern corresponds with a region on the specimen encompassed by a selector aperture (SAD aperture) (fig. 5A). Using this aperture, one can readily obtain the diffraction pattern only from an interesting part rather than the entire field of view. This technique is known as the '*selected-area*

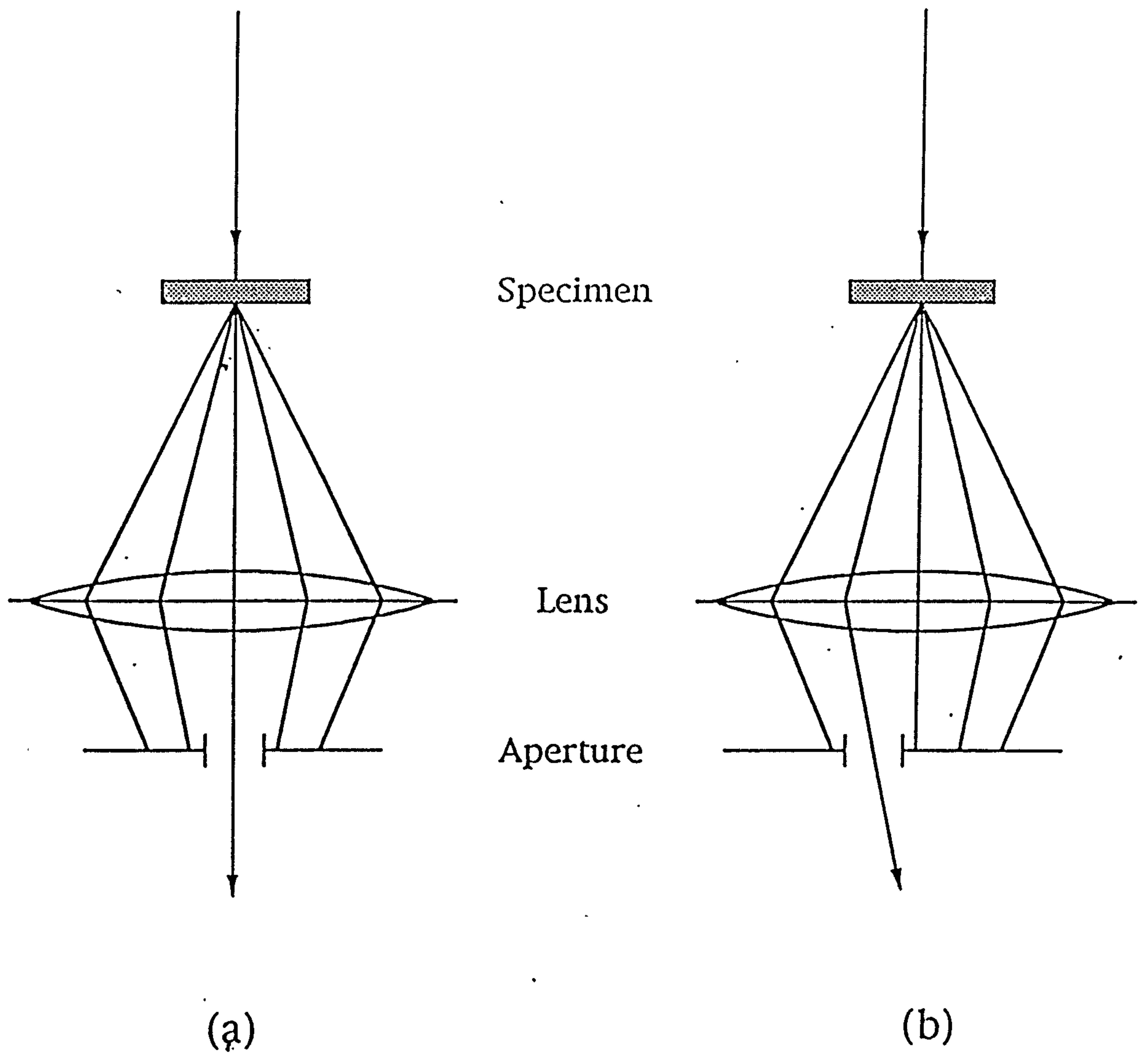


Fig.5B Two models of image formation. (a) Bright-field image. (b) Dark-field image.

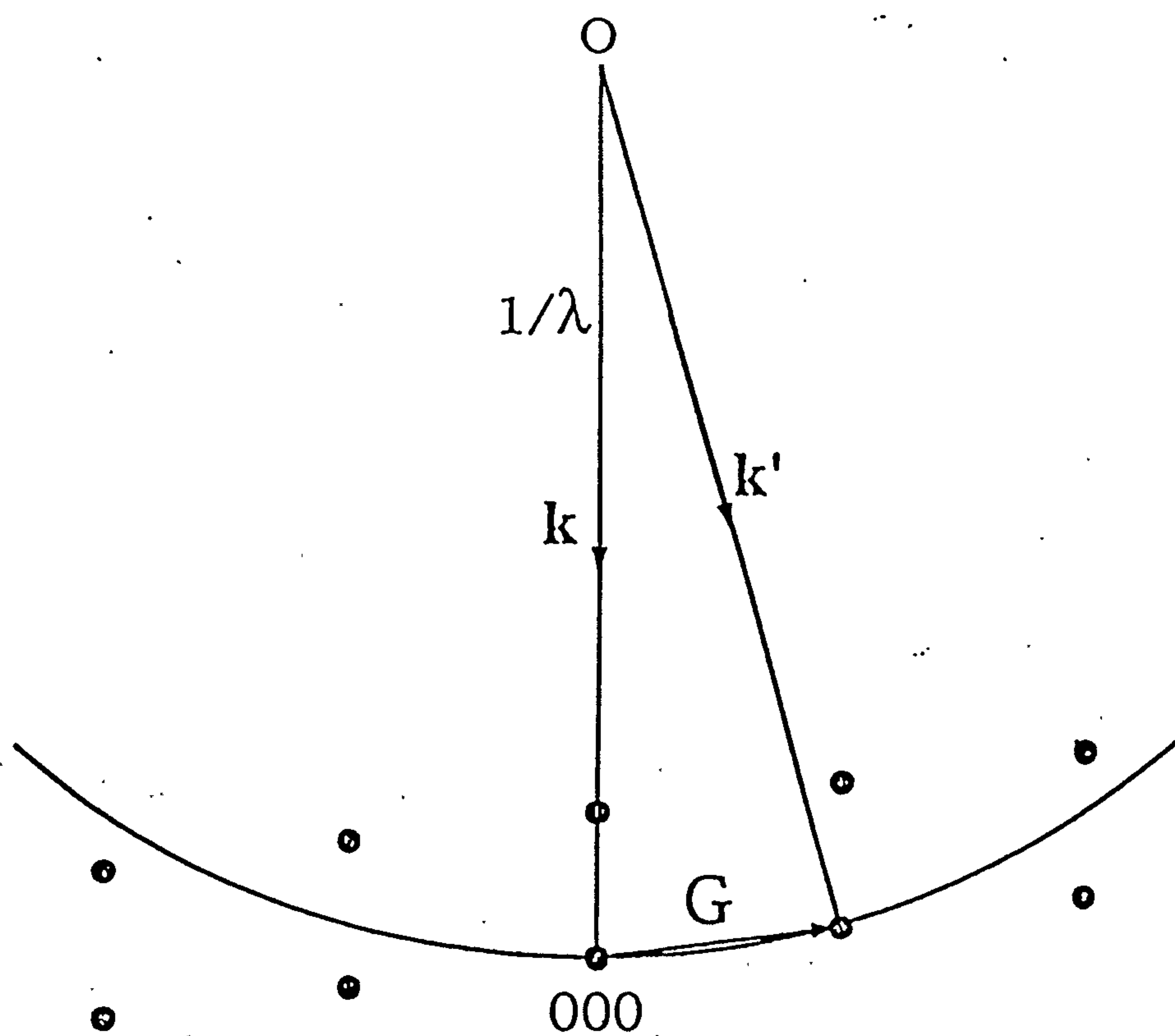


Fig. 5C Reflecting sphere construction.

electron diffraction'. The typical sizes of the SAD apertures for a transmission electron microscope are from 10 to 800 μm in diameter which corresponds to a selected area of a few 0.1 μm to a few μm in diameter on the investigated sample.

The spot recorded in the diffraction pattern correlates with the parallel lattice planes with plane-normal parallel to the reciprocal vector of the given Bragg reflection. If we define the distance of this given reflection from the central spot as R , the interplanar spacing of associated parallel planes in the real space as d , the relation between R and d is then written as Bragg formula:

$$Rd = L\lambda$$

where λ is the electron wave length and L is the camera length defined as the distance of the specimen from the photo plate. $L\lambda$ is called camera constant which can be calibrated by using a known structure [5.26].

5.2.1.4. Energy-dispersive X-ray (EDX) spectroscopy [5.27-5.29, 5.33].

The interaction between the electron beam and a thin foil specimen is very complex and results in various radiations and physical effects (fig. 5D). Among them, the production of characteristic X-rays is now widely used in analytical electron microscopes for identification of elements and determination of compositions. The technique is called energy-dispersive X-ray spectroscopy (EDX). The production of the characteristic X-rays is due to the inelastic interaction between the high energy incident electron beam with the specimen, causing inner shell ionisations of the atoms in the sample [5.28, 5.29]. For example, the ejection of a K shell electron leaves the atom in an excited state. An electron of an outer shell will fall to the vacant inner

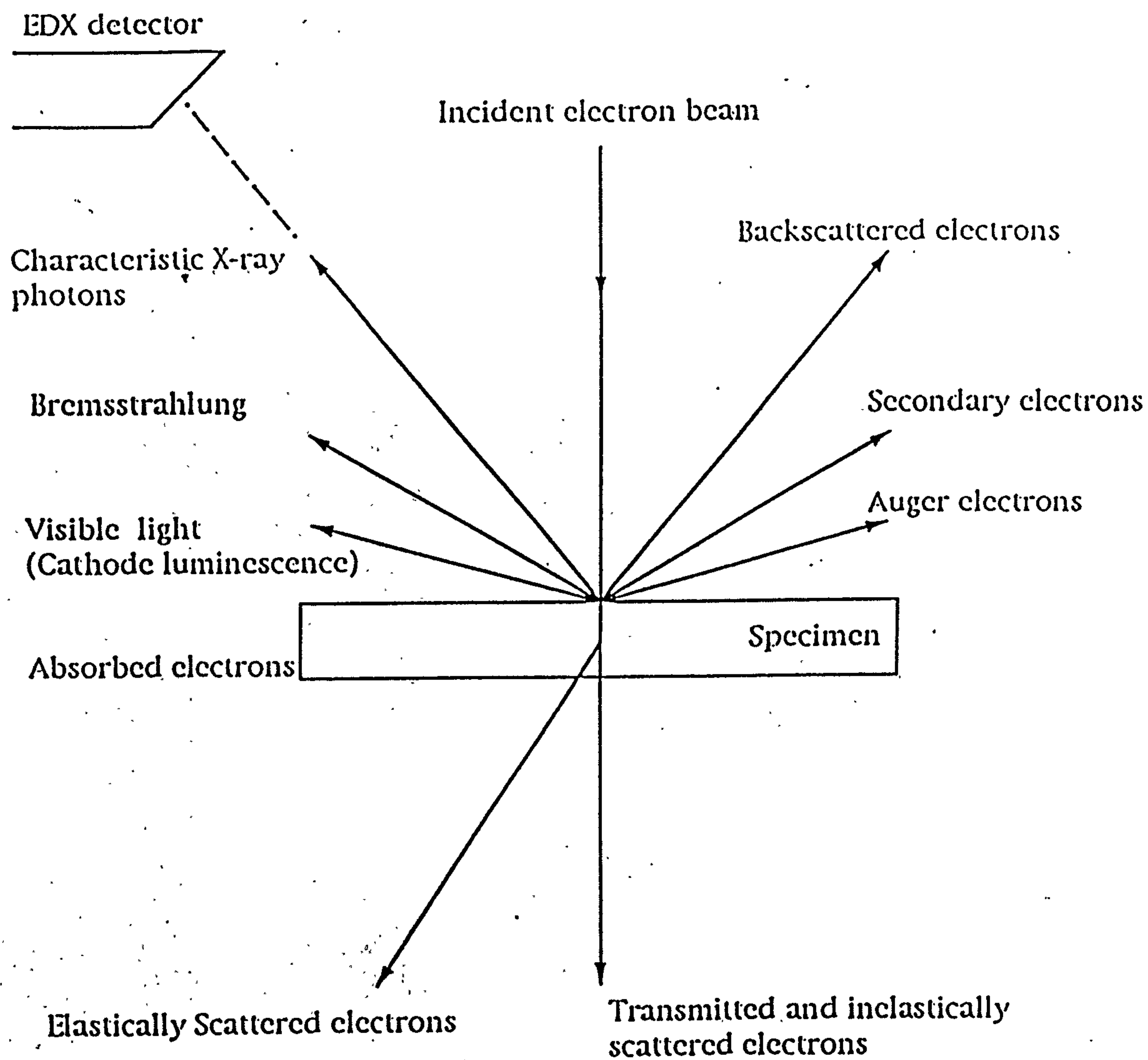


Fig. 5D Various productions due to the interaction of electron beam with a sample.

shell position to lower the potential energy. A simultaneous emission of an x-ray takes place, of which the characteristic energy is determined by and compared with the difference in electron energy levels of all atoms and therefore provides direct chemical information.

In the Philips CM12 transmission electron microscope used in this work, EDS spectra are acquired by using a convergent incident beam, with an area on the sample ranging from 20~100 nm. The acquisition period can be chosen so that more than 10^5 counts are accumulated which is essential to minimise the statistical error [5.27].

5.2.2. Materials:

TEM investigations were carried out on two different brands of orthodontic wire. (See chapter 2 for details)

Wilcock's wires with diameters of 0.52 mm(WF) and 1.62 mm(WS).

Dentaurum Wires with diameters of 0.88 mm (GF) and 2.18 mm (GS).

5.2.3. TEM sample preparation:

Samples for the 0.52mm and 0.88mm diameter wires were prepared in the following way:

1. Short lengths of wire were removed from the spool and cut into lengths of about 1 cm;
2. These lengths of wire were put into a brass tube of 3mm outer diameter and glued in with GATAN G-1 type epoxy; and
3. The tube was sawed to a thickness of about 1 mm.

In this way, a 3mm diameter disc containing a transverse section of the wires was obtained. The longitudinal section of the wires was obtained by sawing the tube in a longitudinal direction to a plate of about 1 mm thickness and then cutting the plate into a 3mm diameter disc.

The procedure for thinning this disc to a TEM sample was done mechanically as far as possible. Afterwards, either ion beam thinning or electropolishing were used.

Unfortunately, the wire's films always separated away from the GATAN G-1 type epoxy when the disc was ground on diamond paper. The best solution found to overcome this problem was to pick up the separated wire films from the diamond paper using tweezers. These separated wire films were already thinned to about ten to twenty micrometers and some parts were even thinner.

These thinner part meant that this part had already been thinned to as small a thickness as the rim of the hole which the electron beam can penetrate. The wire film was then glued to a 3mm outer diameter brass ring and put into an ion beam thinner for one hour each side to clean off surface contamination. In this way, TEM samples of cross section and longitudinal section of 0.52mm and 0.88mm diameter wires were produced.

For the 1.62mm (WS) and 2.18 mm (GS) diameter cross sections and longitudinal sections wires, the TEM sample are prepared by chemical polishing. The chemical solution is 5% HClO_4 in methanol, and the chemical polishing condition are as follows:

Temperature: -70°C ,

Pump setting: 5.8,

Volts: 90 V, and

Current: 50 mA

5.2.4. TEM method:

A Philips CM12 transmission electron microscope (TEM) operating at 120kV was used to analyse the microstructure of the wires. The specimen holder used here was a double tilt holder. The particular TEM technology used was the bright-field and dark-field images, selected area electron diffraction (SAD) and ring diffraction patterns.

5.3. Results:

5.3.1. Grains or subgrains shape and size.

It is important to analyse the shapes of grains or subgrains in the wires, because these can have an important influence on the properties of wires because they introduce anisotropic property. The size of grains or subgrains can also have an important influence on the mechanical properties of the wires as smaller size grains or subgrains have more grain or subgrain boundary area which can hinder the motion of dislocations. More grain or subgrain boundaries also have higher dislocation density to work-harden the structure. [5.30, 5.31, 5.32]. A series of experiments was carried out to investigate the shape and size of the grain or subgrain in WS, WF, GS and GF wires.

In the following, we will see that GS wires have the hexagonal grains as this wires' structure have recovered from the heat-treatment. It is however, the

grains in WF, WS and GF wires are seriously deformed. All the grains in this kind of structure are not grains and so they are called subgrains. The subgrains in these three wires are extremely elongated. The measurement of the width of the subgrains and grains is performed by counting the number of the subgrains' widths and grains' diameters within a given length. This counting is done by determining the number of subgrains or grains that intersect a given length of line.

Fig 5.1~5.7 shows the bright field images and dark field images in longitudinal section of the WS, WF, GS and GF wires. It is evident that the subgrains are elongated in WS, WF and GF wires (fig.5.1, 5.3 & 5.6). The measurements of the average width of these subgrains by using the dark field images (fig.5.2, 5.4 & 5.7) are shown in the table 5.1. The GS wire has a hexagonal shaped grain (fig.5.5), which is very different in comparison with the other wires. The measurements of average subgrains or grains size of the longitudinal section is also shown in table 5.1.

Table.5.1.

Wires	WS (um)	WF (um)	GS (um)	GF (um)
Figures No.	Fig.5.1	Fig.5.3	Fig.5.5	Fig.5.6
Ave. subgrains width or grains size	0.23±0.03,	0.030±0.005	12.00±0.10	0.110±0.015

The reason we talk of "subgrains" in the WS, WF and GF wires is because the original grains in these wires are seriously deformed into subgrains through the heavy wire drawing process. GS wires still have hexagonal shape grains, probably due to high temperature annealing recovering the deformed subgrains into grains.

The grain or subgrain shape and size in the cross section of WS, WF, GS and GF wire were investigated using TEM as shown in figures 5.8 ~ 5.18. The measurements of grains or subgrains size of above wires in cross section are shown in the table.5.2.

Table. 5.2:

	Ave. length of subgrains or grains in the core(um).	Ave. width of subgrains or grains in the core(um).	Ratios of length/width	Ave. length of subgrains or grains in the shell(um).	Ave. width of subgrains or grains in the shell(um).	Ratios of length/width	Figures No.
WS	0.250 $\pm$ 0.030	0.180 $\pm$ 0.030	1.5	0.450 $\pm$ 0.030	0.220 $\pm$ 0.030	2	5.8, 5.9 & 5.10
WF	0.040 $\pm$ 0.005	0.030 $\pm$ 0.005	1.3	0.130 $\pm$ 0.005	0.018 $\pm$ 0.005	8	5.11,5.12&5.13
GS	12.000 $\pm$ 0.100	12.000 $\pm$ 0.100	1.	12.000 $\pm$ 0.100	12.000 $\pm$ 0.100.	1	5.14 & 5.15
GF	0.110 $\pm$ 0.015	0.110 $\pm$ 0.015	1	0.110 $\pm$ 0.015	0.110 $\pm$ 0.015	1	5.16,5.17&5.18

Table 5.2 and figs. 5.8, 5.9, 5.11 & 5.12 show that for the WS and WF wires, the subgrains in the core are less elongated than in the shell of the wires. For the GF wires (figs. 5.16 & 5.17 and the measurement in table 5.2), the shape and size of subgrains in the shell and in the core of cross section are similar. For the GS wires, the grains in the core and in the shell are both hexagonal



fig. 5.1



fig.5.2



fig. 5.3



fig. 5.4

Fig.5.1 BF images taken from the core of the WS wires longitudinal section samples. The subgrains shape is clearly elongated.

Fig.5.2 DF image taken from the core of the WS wires longitudinal section samples. The average width of the subgrains is calculated as about 0.23μm.

Fig.5.3 BF images taken from the WF wire longitudinal section samples. The subgrains shape is clearly elongated.

Fig.5.4 DF image taken from the core of the WF wires longitudinal section samples. The average width of the subgrains is calculated as about 0.03μm..



fig.5.5



fig.5.6



fig. 5.7

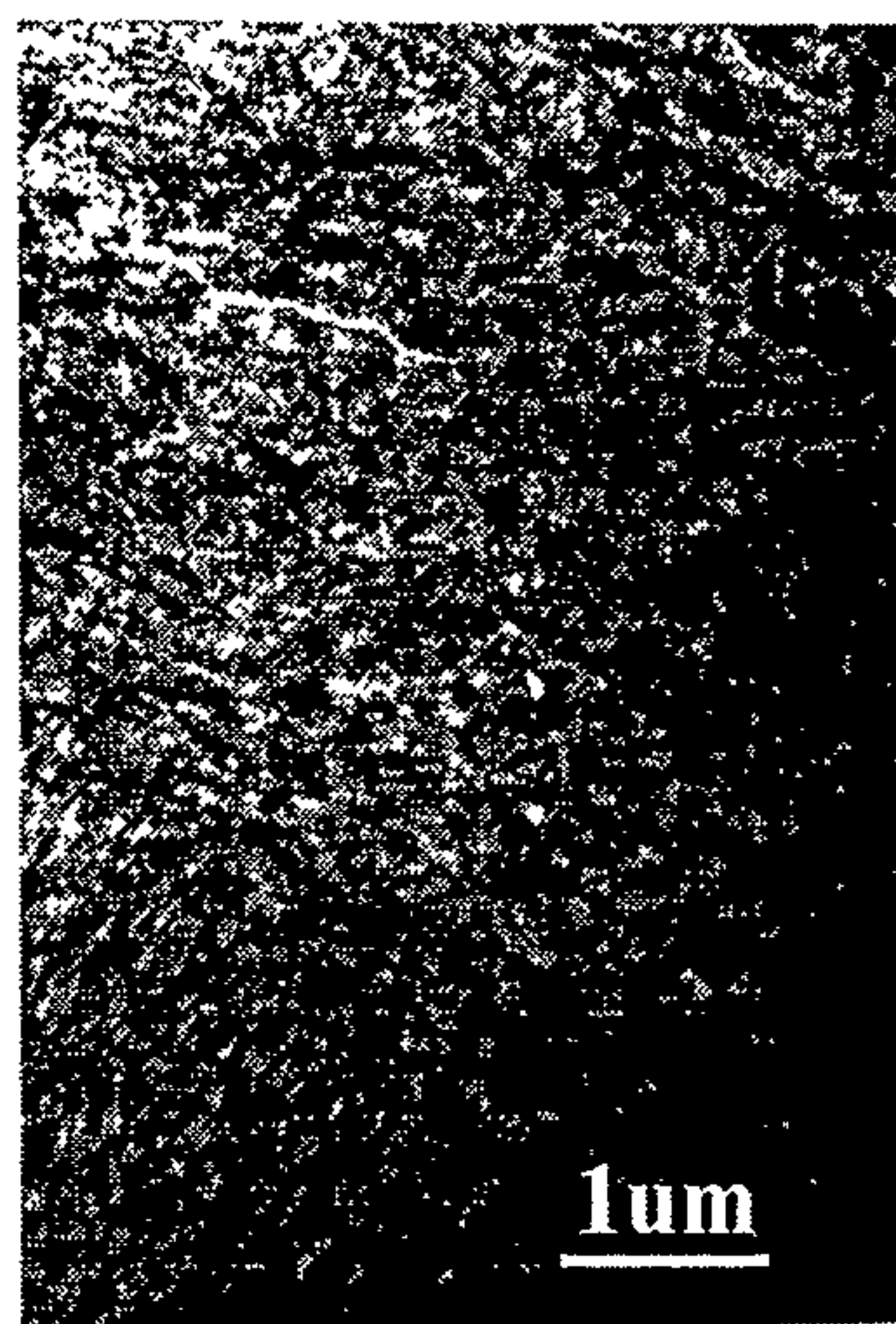


fig. 5.8

Fig.5.5 BF images taken from the core of the GS wires longitudinal section samples. The grain is hexagonal shape. The average grain size is calculated as about 12.00μm.

Fig.5.6 BF image taken from the corn of the GF wire longitudinal section samples. The subgrains shape is clearly elongated.

Fig.5.7 DF image taken from the core of the GF wires longitudinal section samples. The average width of the subgrains is calculated as about 0.11 μm.

Fig.5.8 BF image taken from the core of WS wires cross section sample.



fig.5.9

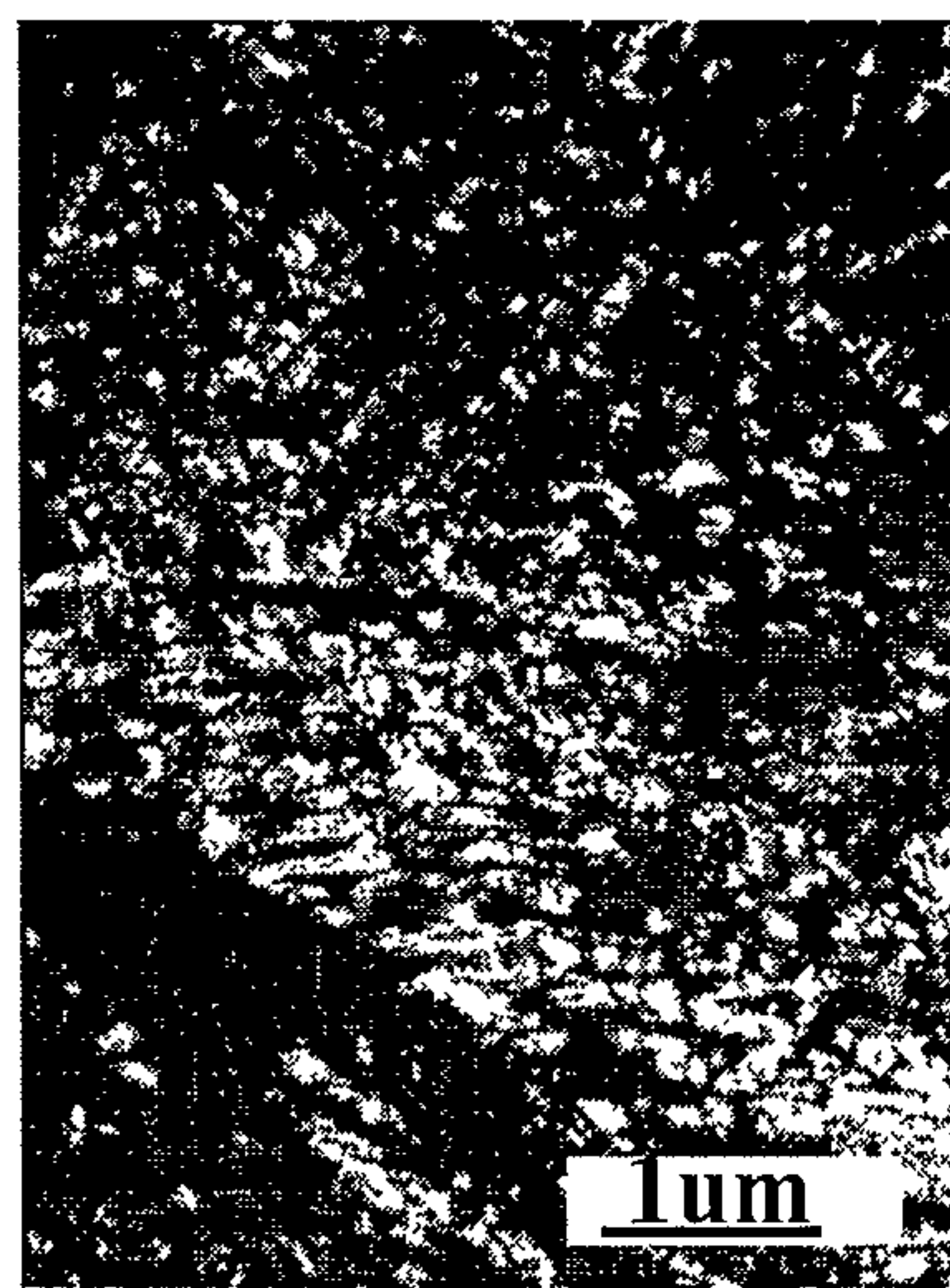


fig. 5.10

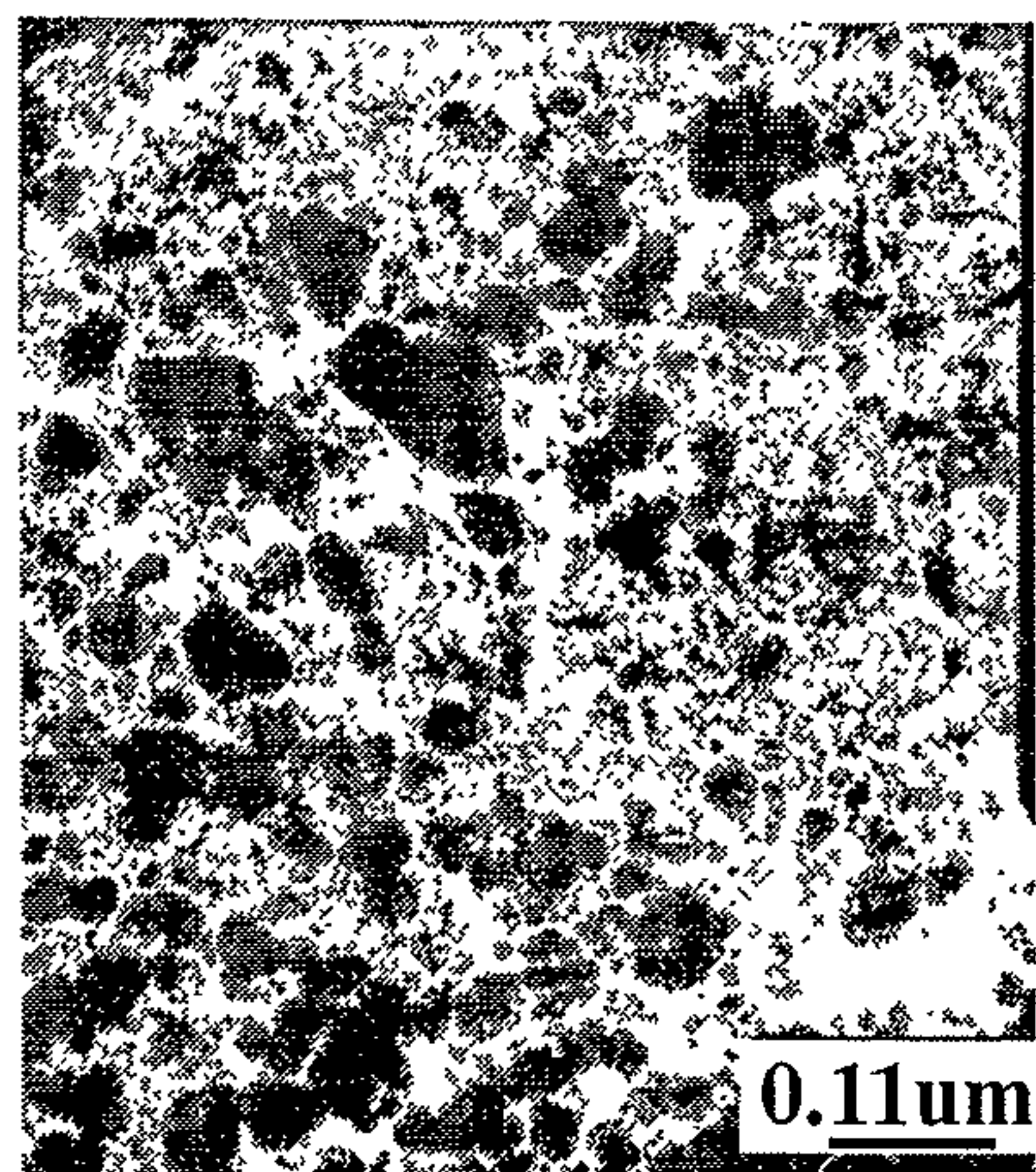


fig. 5.11

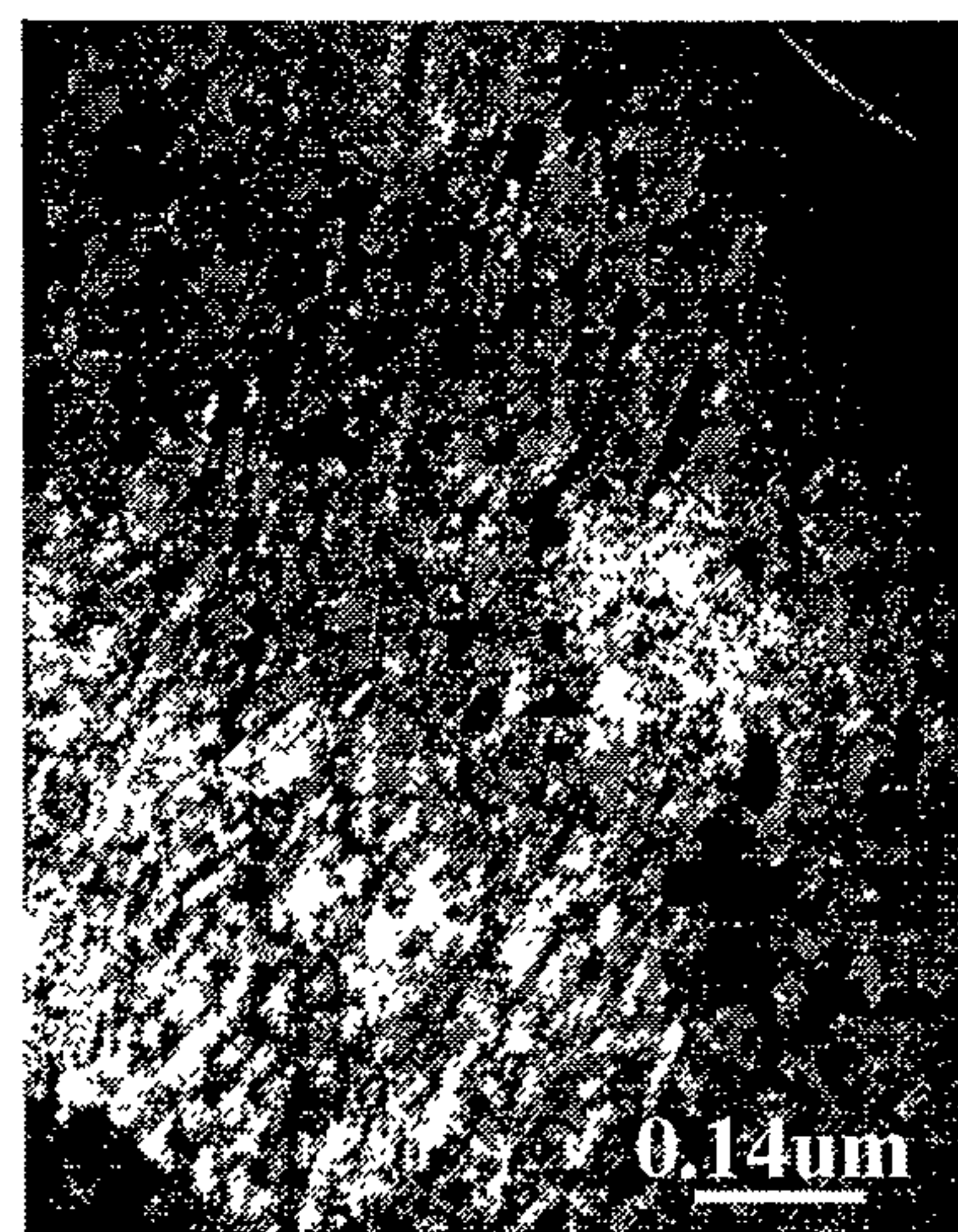


fig. 5.12

Fig. 5.9 BF image taken from the shell of WS wires cross section sample.

Fig.5.10 DF image taken from the core of WS wires cross-section sample.
The average subgrains size was calculated as about 0.23μm.

Fig.5.11 BF image taken from the corn of the WF wires cross section sample.

Fig.5.12 BF image taken from the shell of the WF wires cross section sample.



fig.5.13



fig.5.14

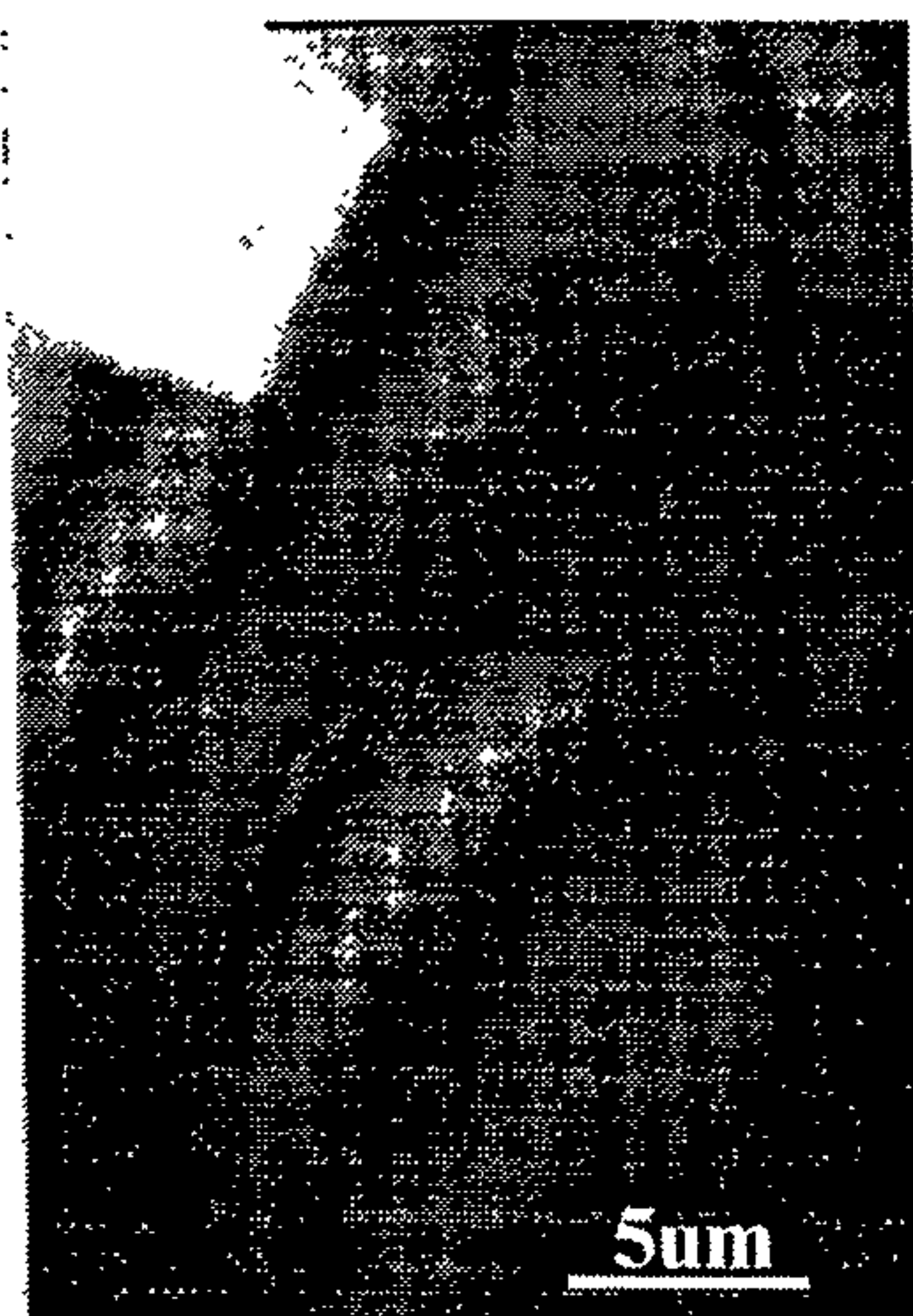


fig.5.15

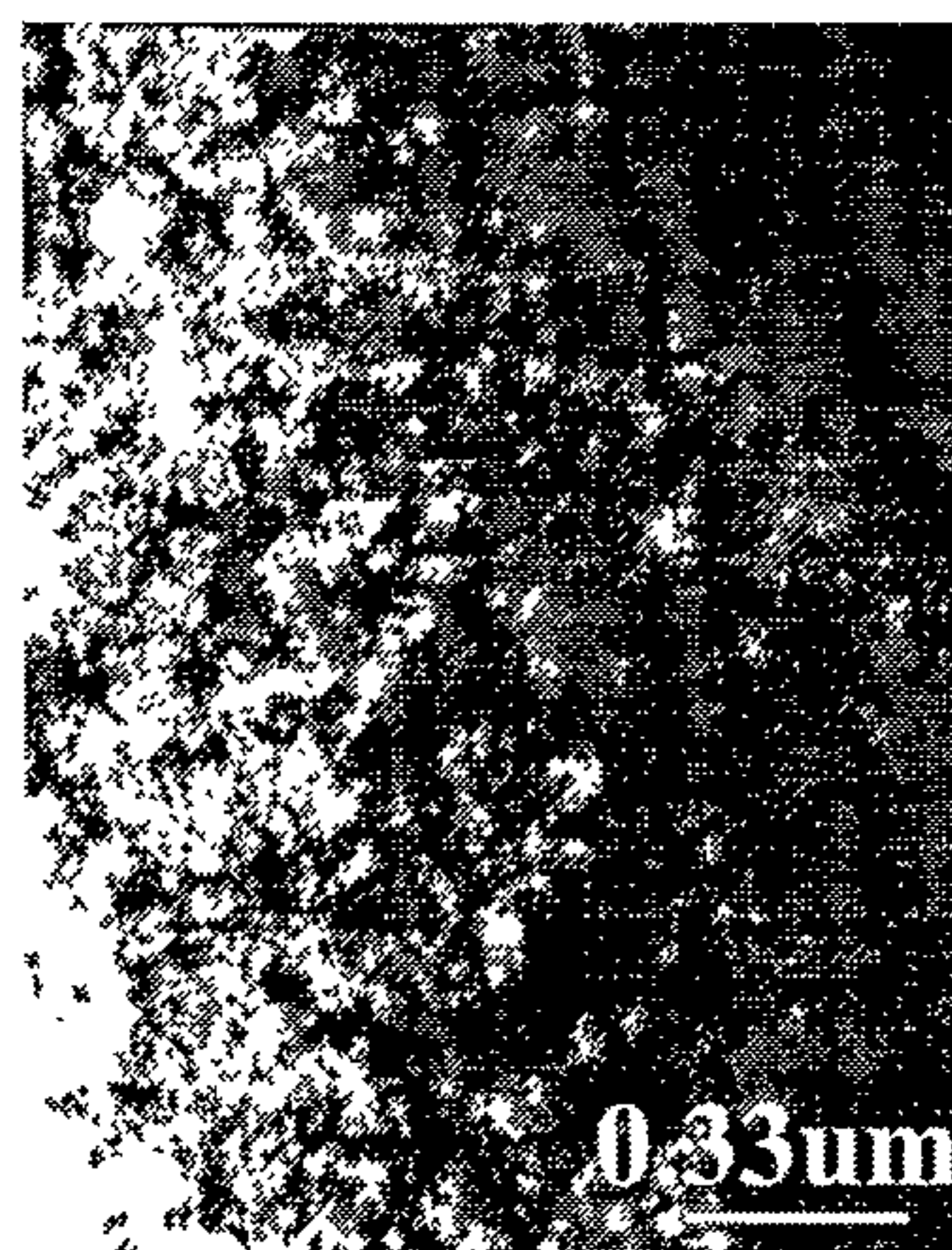


fig. 5.16

Fig.5.13 DF image taken from the core of the WF wires cross section samples. The average subgrains size are calculated as about 0.03μm

Fig.5.14 BF image taken from the corn of GS wire cross section sample. The grains shape are hexagonal. The average grain size measured is about 12μm.

Fig.5.15 DF image taken from the shell of GS wire cross section sample. The grains shape are hexagonal.

Fig.5.16 BF image taken from the core of the GF wire cross section sample.

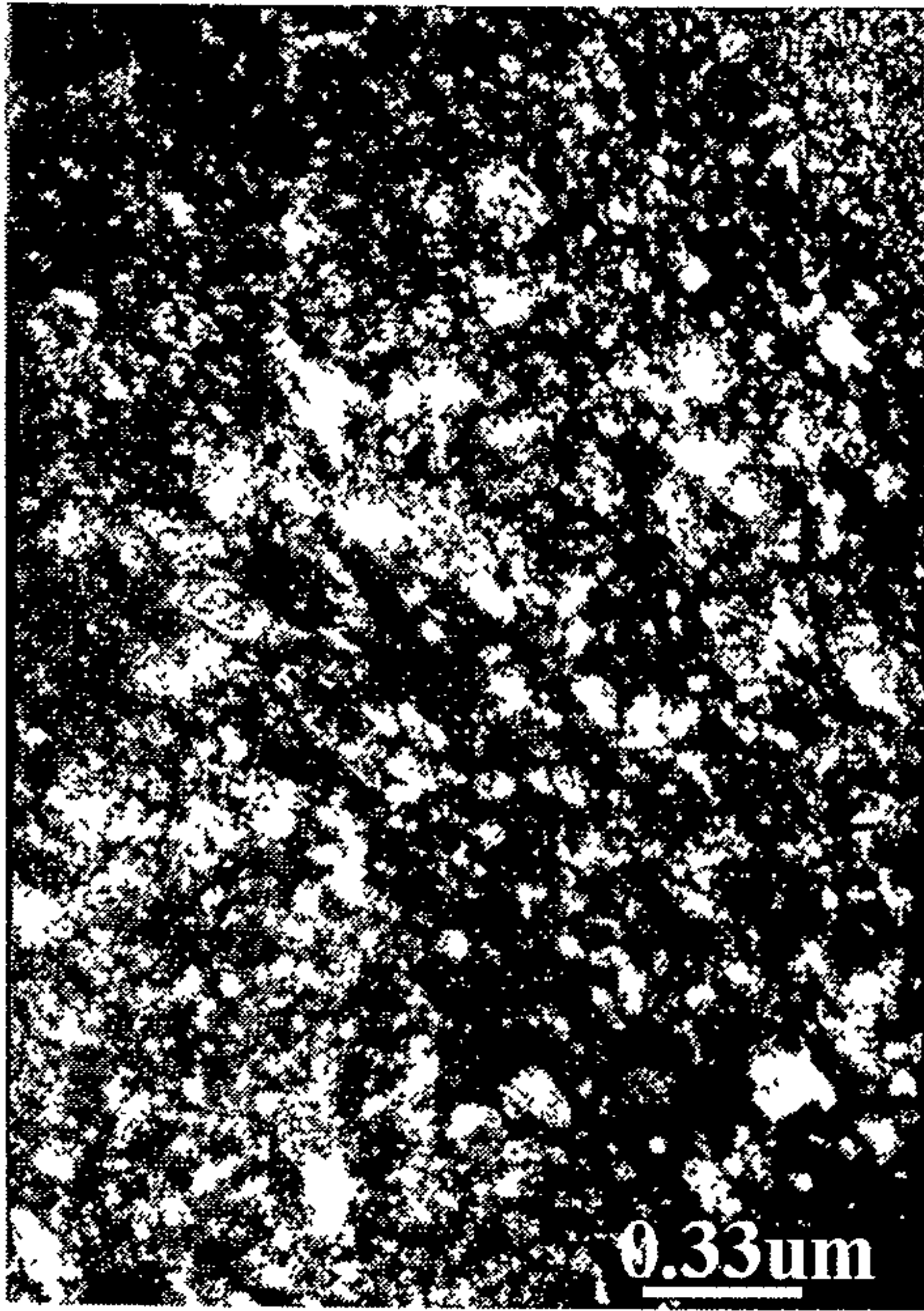


fig. 5.17

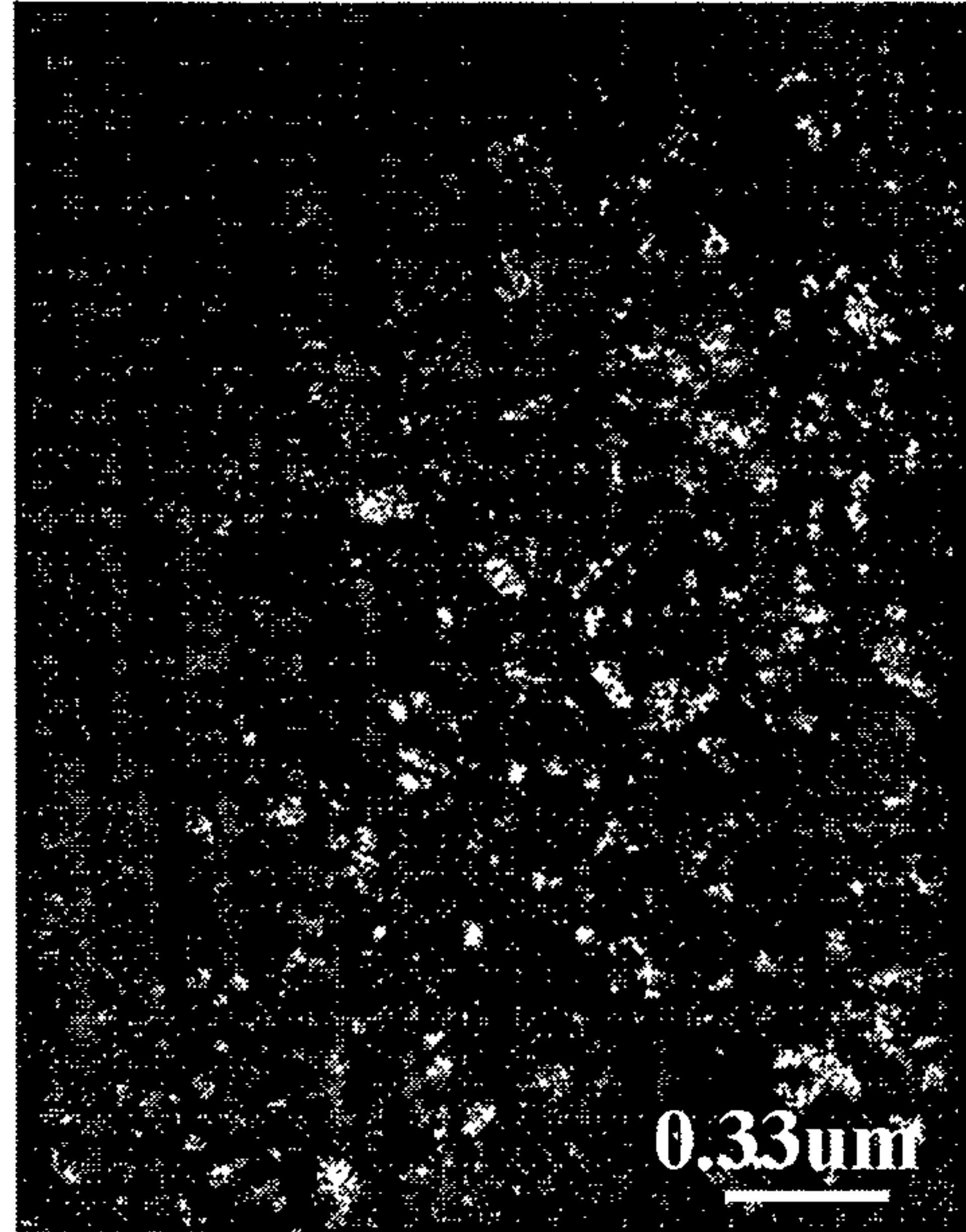


fig. 5.18

Fig.5.17 BF image taken from the shell of the GF wires cross section sample.

Fig.5.18 DF image taken from the core of the GF wires. The subgrains size measured are about 0.10μm.

shape with similar grain size (fig.5.14 & 5. 15 and the measurement in table 5.2). The reason for different shape of grains and subgrains between the shell and core is due to the different manufacture process. These results for the grain or subgrain size in the core of cross sections of WS,WF,GS and GF wires are consistent with the average width of grains or subgrains in the core of longitudinal sections of the same wires.

5.3.2. The phases present in the WS,WF,GS and GF wires and their microstructure morphology.

It is important to analyse the different phases and their microstructure in the wires because these have a great influence on the mechanical properties of the wires. The microstructure such as phases, texture, twinning and small angle twist boundaries result from the level of structure deformation and influence the strength of the wires.

5.3.2.1. Ring pattern

Electron diffraction can be used to analyse the texture, the phases in polycrystalline material. The ring patterns for WS, WF and GF wires are shown in figs.5.19, 5.20 and 5.21 respectively. The interplanar spaces of these rings are shown in table 5.3, and it seen that, for each case, they are consistent with a mixture of martensite and austenite.(figs. 5.19~5.21', & table 5.3.) The calculated average lattice parameters for austenite phases in WS, WF and GF wires is $3.71 \pm 0.05\text{\AA}$, $3.73 \pm 0.07\text{\AA}$, and $3.72 \pm 0.08\text{\AA}$, and $2.93 \pm 0.06\text{\AA}$; $2.91 \pm 0.06\text{\AA}$; $2.90 \pm 0.04\text{\AA}$ for the martensite phase. These values agree well with to *the standard austenitic phase (fcc) lattice parameter of $3.65\text{\AA}$ and α -martensitic phase (bcc) lattice parameter of*

2.87A. Thus, the lattice parameter calculations indicate that both austenitic and martensitic phases are present in the WS, WF and GF wires.

The fact that all these rings are incomplete, and in the form of arcs in figs.5.19, 5.20 & 5.21 shows that both the austenitic and martensite phases are textured [5.33].

For the GS wires, the diffraction pattern is not in the form of rings since this wire is not polycrystalline.

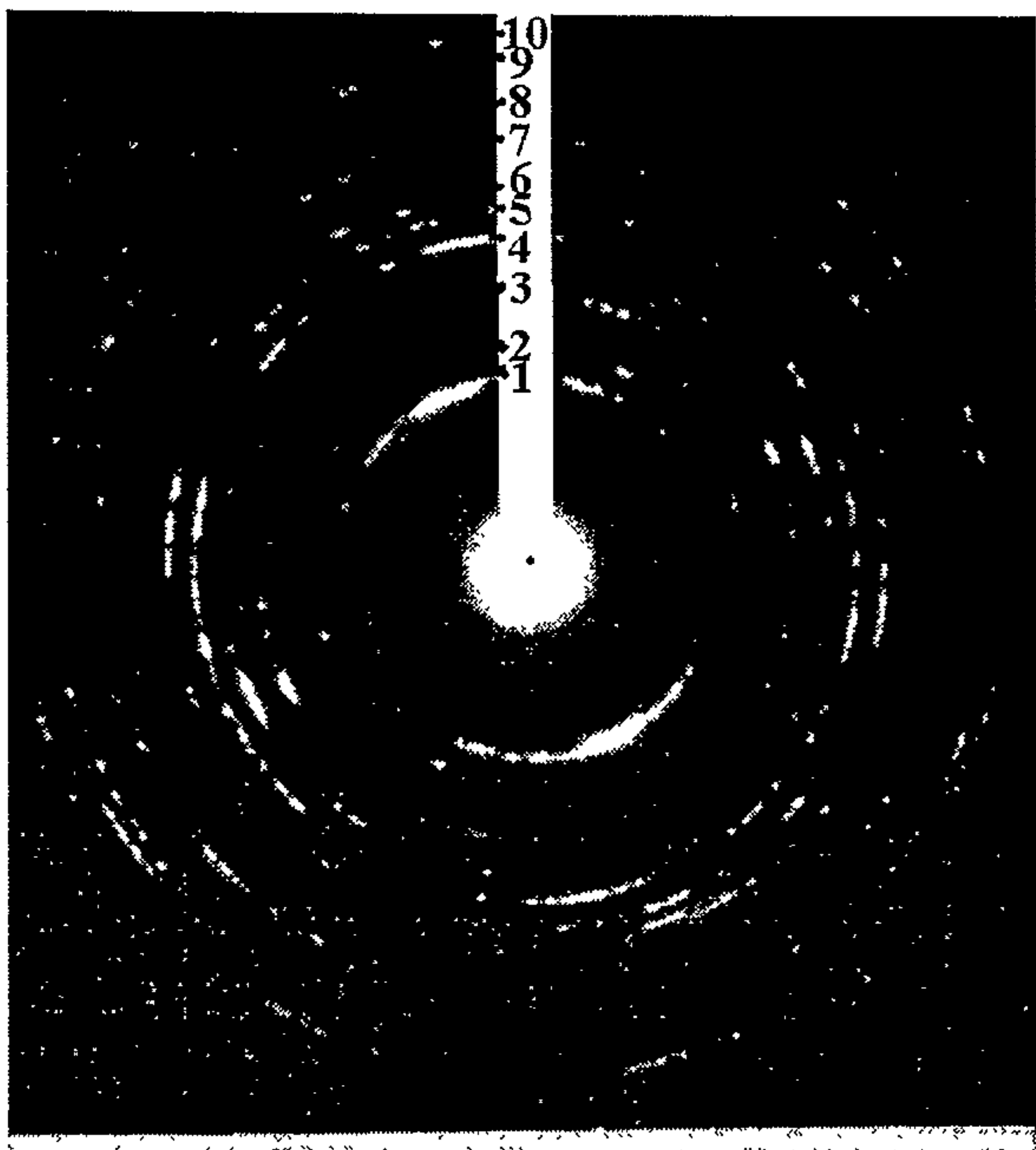


fig.5.19



fig.5.20

Fig.5.19 Ring diffraction pattern taken from the cross section of the wires. The calculated results indicate austenitic and martensitic phases are present in the WS wires. The fact that all these rings became arcs shows that both the austenitic and martensite phases are textured.

Fig.5.20 Ring diffraction pattern taken from the cross section of the WF wires. The calculated results indicate austenitic and martensitic phases are present in the WF wires. The fact that all these rings became arcs shows that both the austenitic and martensite phases are textured.



fig. 5.21

Fig.5.21 Ring diffraction pattern taken from the cross section of the GF wires. The calculated results indicating the structure is composed of many ultra fine grains and contains both austenitic and martensitic phase. Both the austenitic and martensitic phases shows texture structure.

Table 5.3: The calculated ring pattern for WS, WF and GF wires.

WS						
No.	D(mm)	d	N(fcc)	a(fcc)	N(bcc)	a(bcc)
1	22.9	2.13	3	3.69	2	3.02
2	26.5	1.84	4	3.69		
3	33	1.48			4	2.96
4	38	1.29	8	3.64		
5	41.5	1.18	11	3.90	6	2.88
6	45	1.09	12	3.76		
7	47	1.04			8	2.94
8	53	0.92	16	3.69	10	2.91
9	58	0.84			12	2.92
10	60	0.81	20	3.64		
			Average: 3.71		Average: 2.93	
WF						
1	22.85	2.14	3	3.70	2	3.02
2	26	1.88	4	3.76		
3	33.5	1.46			4	2.92
4	37.5	1.30	8	3.68		
5	41.5	1.18	11	3.90	6	2.88
6	44.5	1.10	12	3.80		
7	48	1.02			8	2.88
8	53	0.92	16	3.69	10	2.91
9	59	0.83			12	2.87
10	60	0.81	20	3.64		
			Average: 3.73		Average: 2.91	
GF						
1	22.8	2.14	3	3.71	2	3.03
2	26.5	1.84	4	3.69		
3	34	1.44			4	2.87
4	37.5	1.30	8	3.68		
5	41.5	1.18	11	3.90	6	2.88
6	45	1.09	12	3.76		
7	48.5	1.01			8	2.85
8	53	0.92	16	3.69	10	2.91
9	59	0.83			12	2.87
10	60	0.81	20	3.64		
			Average: 3.72		Average: 2.9	

D: Ring diameter; d: Interplanar spacing; N: Crystal face; a: Lattice parameter.

5.3.2.2. Selected area diffraction, bright and dark field image.

Three TEM techniques-selected area diffraction (SAD), bright and dark field (BF and DF) were used to further analyse the present phases and the morphology of the WS, WF, GS and GF wires.

5.3.2.2.1. WS wires:

As I mentioned in last section, the standard austenitic phase lattice parameter is 3.65Å and martensitic phase lattice parameter is 2.87Å.

The microstructure morphology of the longitudinal section WS wires is shown in fig.5.22. The observed band structures in fig.5.22 can be identified as the austenitic twin structures in the SAED pattern (fig.5.23 and illustrated in fig.5.23'). The diffraction pattern in fig.5.23 can be separated into two: $[101]_{fcc}$ and $[101]_{fcc}$ as shown in fig.5.23'. The calculated lattice parameters for both patterns are $3.62 \pm 0.3\text{Å}$. These values correspond to the austenitic phase lattice parameters of 3.65Å.

Thus, we can conclude that the diffraction pattern in fig.5.23 shows the band structure to be an austenitic twinning structure. The austenitic twin zone axis is $[101]$ and the twin plane is (111).

Figs.5.24, 5.25, and 5.26 are the dark field images obtained using $(020)_t$, $(111)_\gamma$ and $(111)_{\gamma+t}$ diffraction spots respectively for the diffraction patterns shown in fig.5.23. These dark field images show that all the bright bands are due to the twin austenitic structure.

By using a double tilt specimen holder, a series of diffraction patterns was taken tilting the region to different zone axes. Figs.5.27,5.28,5.29 are the

diffraction patterns for the $[001]_{\text{fcc}}$, $[011]_{\text{fcc}}$, and $[112]_{\text{fcc}}$ zones taken from one of these bands (fig.5.22) within the twinned structure. The lattice parameters for these diffraction patterns are $3.64 \pm 0.03 \text{ \AA}$, $3.63 \pm 0.03 \text{ \AA}$ and $3.61 \pm 0.04 \text{ \AA}$, respectively. These values correspond to the austenitic phase lattice parameters. The above SAD patterns further confirm that the austenitic phase exists in the WS wires.

Details of the tilt experiments are in the appendices to this chapter: Appendix 5.1: the SAD diffraction zones and respective scale of X,Y axis of the double tilt holder for figs.5.27, 5.28, and 5.29; Appendix 5.2: the respective experimental and theoretical inter-zones between the zones for figs.5.27, 5.28, and 5.29.

A small angle twist boundary was found in figs.5.30-5.32. The $[112]_{\text{fcc}}$ selected area diffraction pattern of the austenitic phase of (fig.5.30) can be taken from the arrowed grains in the image of fig.5.31 which is taken from the longitudinal section wires. The elongated diffraction spots are due to small angle twist boundaries between individual twin plates. The angle of $(13\bar{1})$ reflection spot in the diffraction pattern is about 6° . This can be confirmed by observing the bright field image in fig.5.31 and the $(13\bar{1})$ dark field image of fig.5.32. The angle we measured between these bands in bright and dark field image is also about 6° .

In the same specimen as considered of above, some parts were found to have martensite. The martensite phase was identified from the diffraction pattern. fig.5.33 shows an example for the region shown in fig.5.34. The calculated lattice parameter is $2.88 \pm 0.03 \text{ \AA}$, which corresponds with the martensite phase lattice parameter of $2.87 \text{ \AA}$. Fig 5.35 is the dark field image taken

using the (110) spot in fig.5.33. This dark field image shows that the martensitic phase looks similar to the austenitic phase described above.

In the TEM image, the martensite and austenite appear similar (bands, twins etc). This similarity is found in both BF and DF images. Fig.5.36 is a diffraction pattern taken from the bright field image of fig.5.37. The diffraction pattern contains two diffraction patterns [001] austenitic phase and [001] martensitic phase as illustrated in fig.5.36'. The calculated lattice parameter for the inner pattern is $2.89 \pm 0.02 \text{ \AA}$ which corresponds to the martensite phase. The calculated lattice parameter for the outer pattern is $3.66 \pm 0.03 \text{ \AA}$ which corresponds to the austenite phase. Fig.5.38 and fig.5.39 are the dark field images taken using the martensite (110) spot and austenite (020) spot, respectively.

These two dark field images tell us again that it is almost impossible to distinguish the austenite and martensite phases through the image in the WS wires since both of them have identical band twinning structures.



fig. 5.22

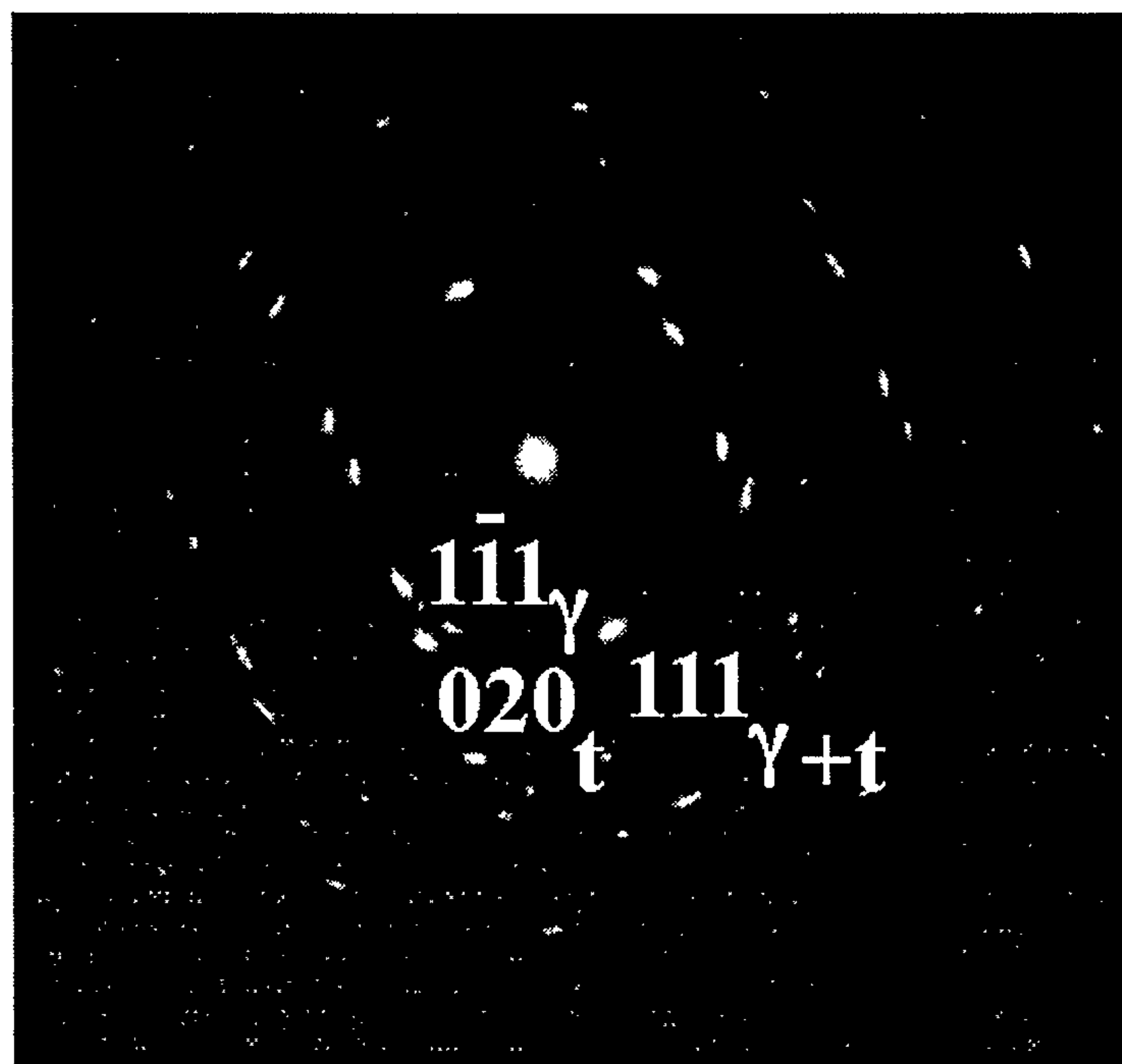


fig. 5.23

Fig.5.22 BF image taken from the longitudinal section of WS wire. It clearly revealing the band structure.

Fig.5.23 SAED pattern taken from the area show in the fig.5. 22 and clearly revealing twinning.

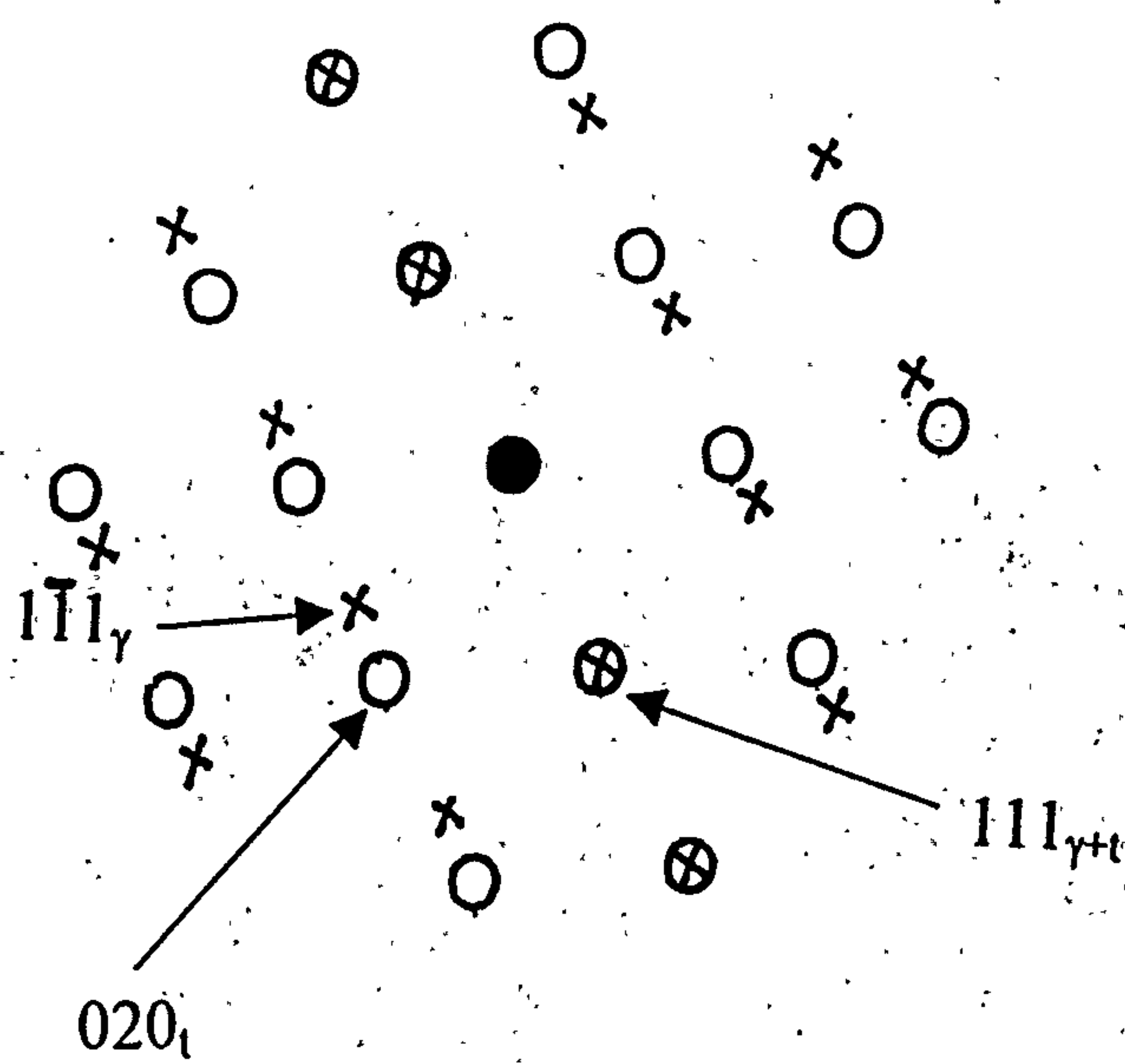


Fig. 5.23'

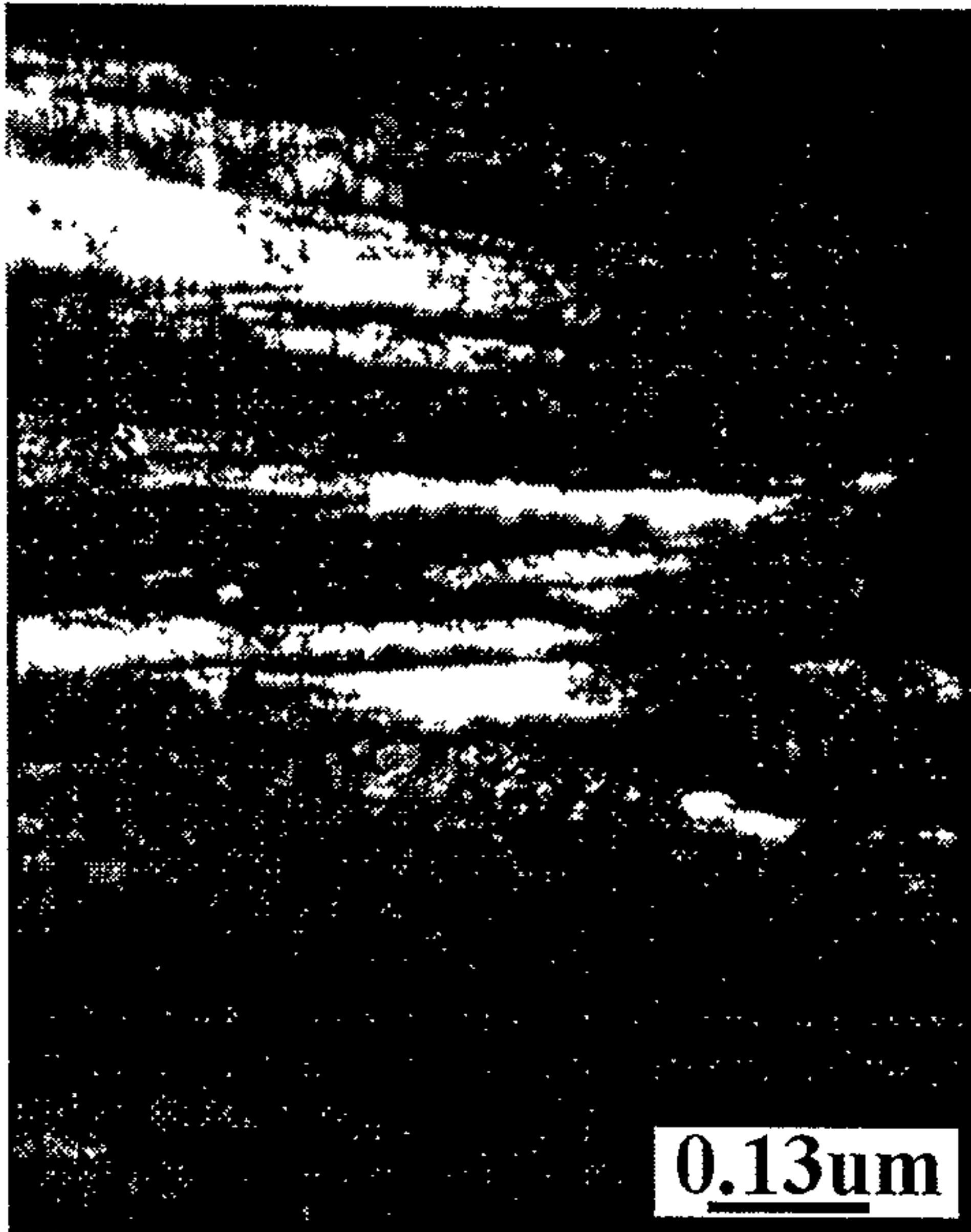


fig. 5.24

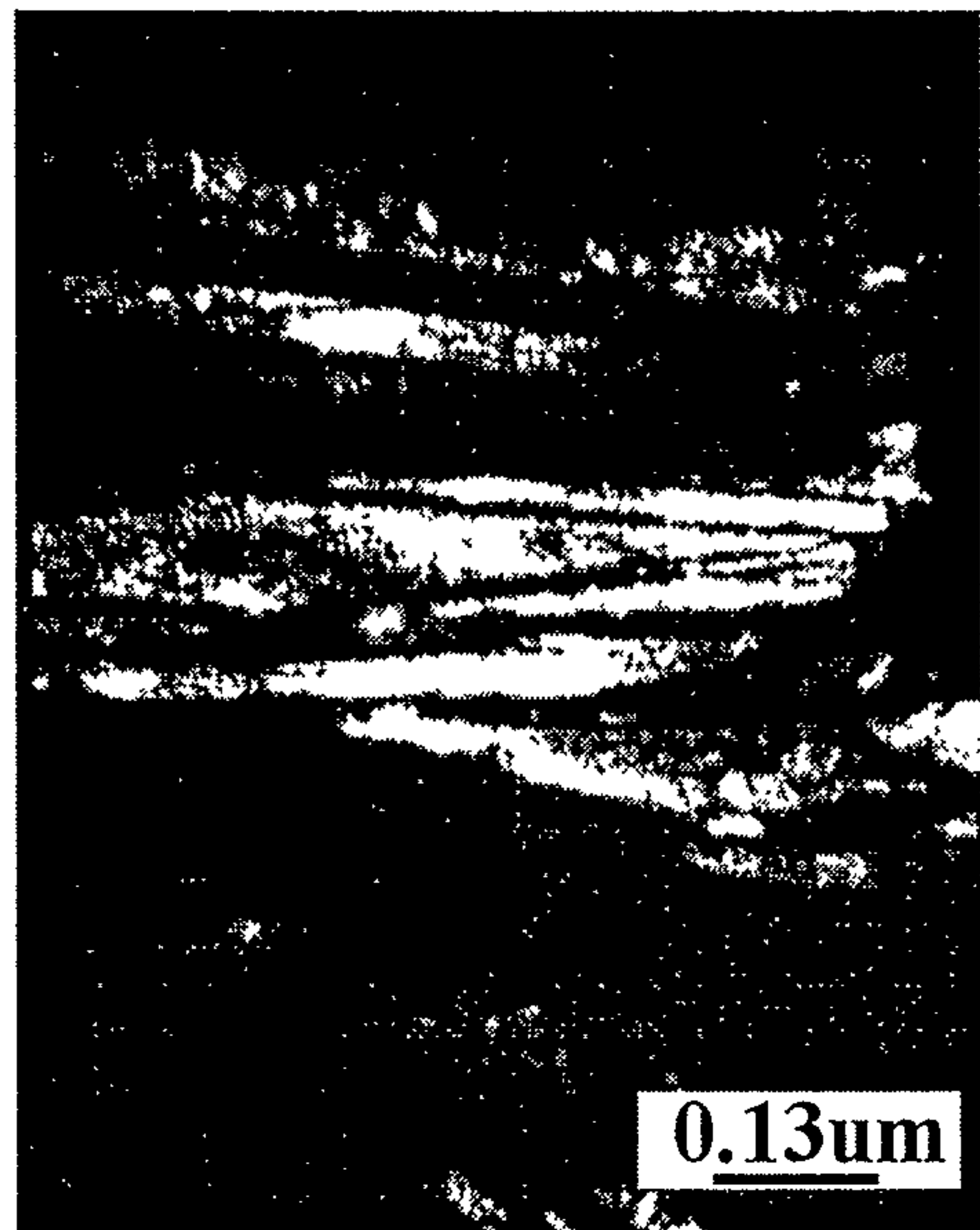


fig. 5.25



fig. 5.26

Fig.5.24 DF image which taken with one of the twinning $(020)_t$ reflections of the austenite phase in fig. 5.23.

Fig.5.25 DF image which taken with one of the twinning $(1\bar{1}1)_\gamma$ reflections of the austenite phase in fig. 5.23.

Fig.5.26 DF image which taken with one of the twinning $(111)_\gamma+t$ reflections of the austenite phase in fig. 5.23.

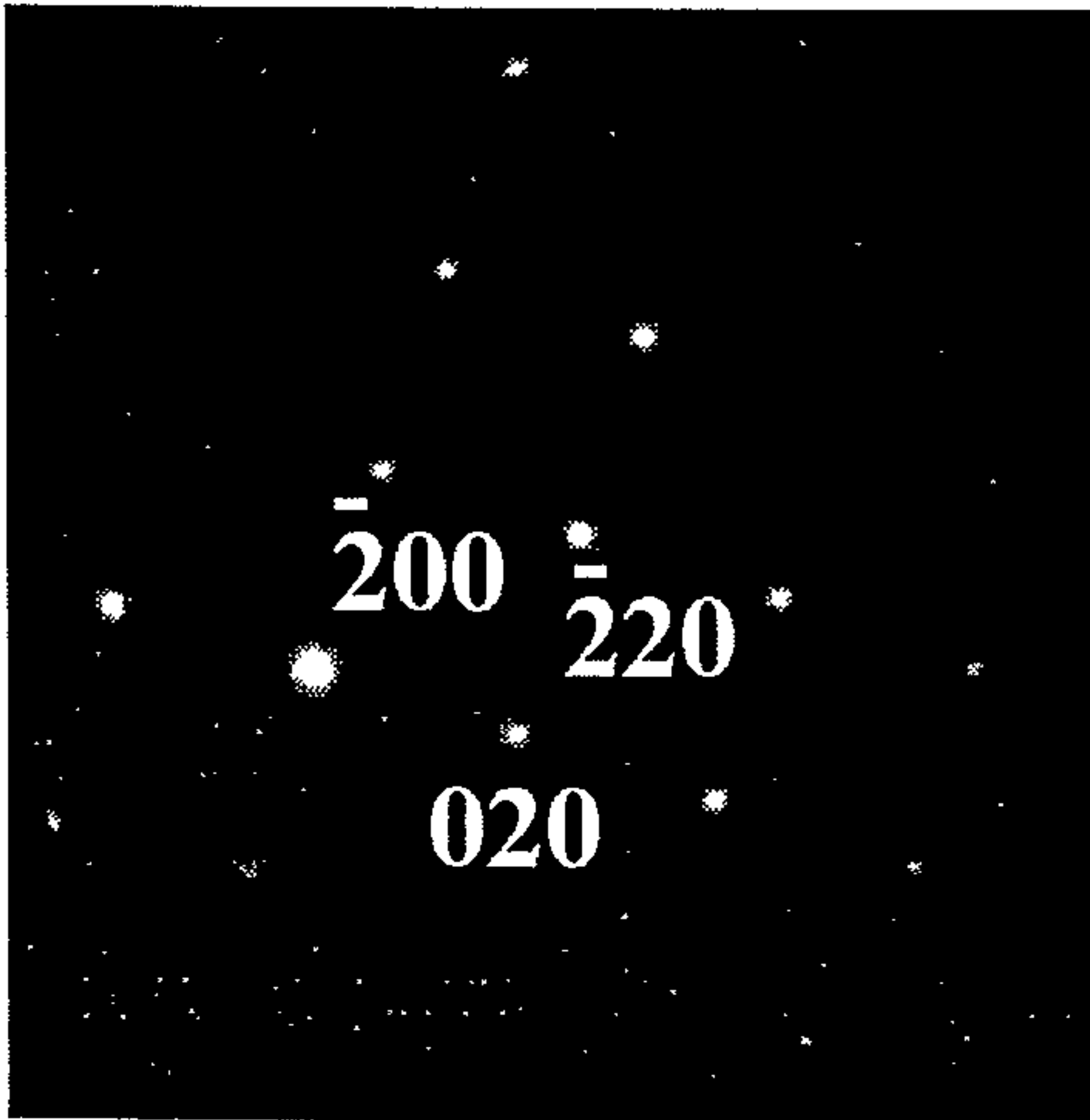


fig. 27

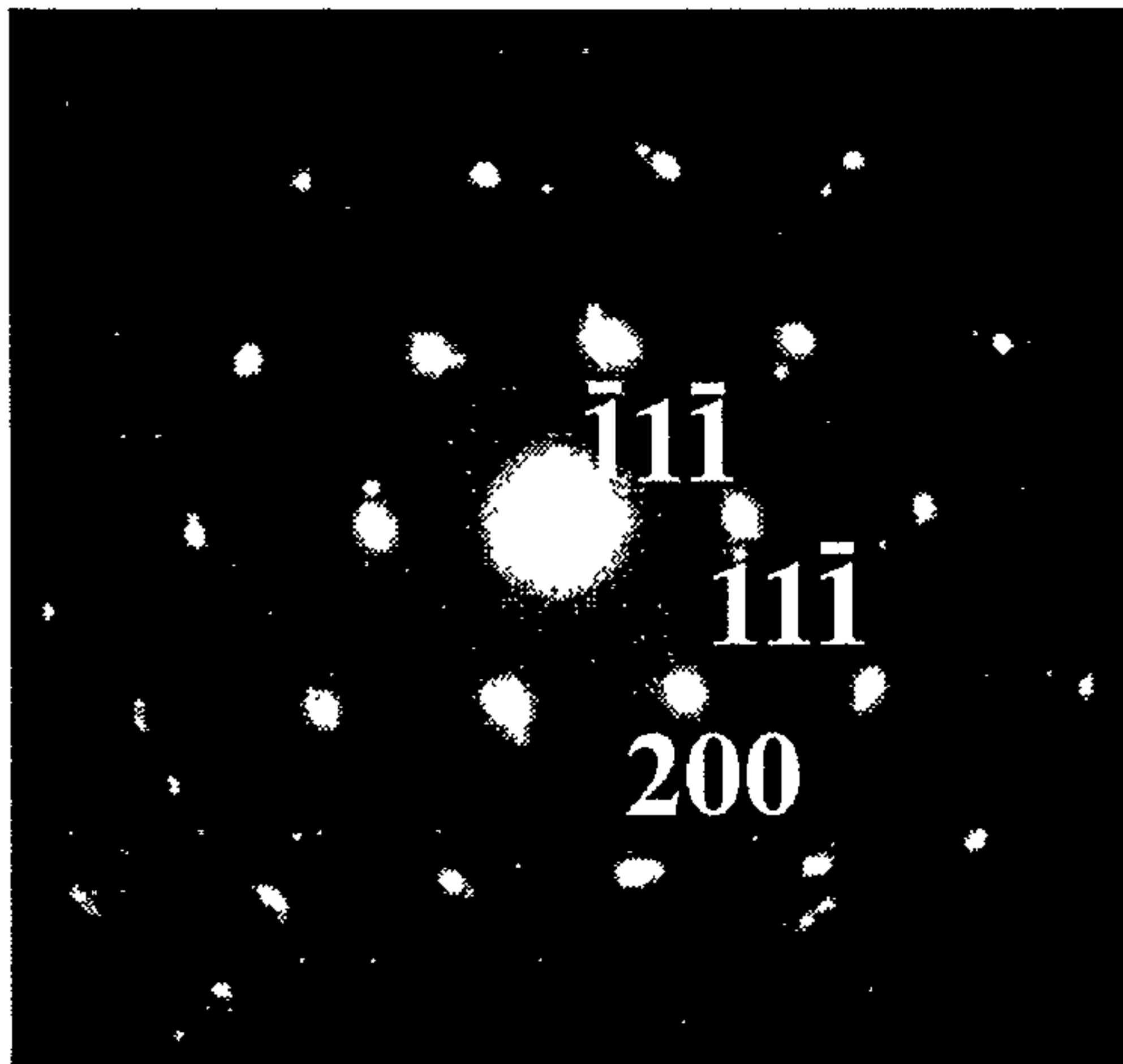


fig. 28

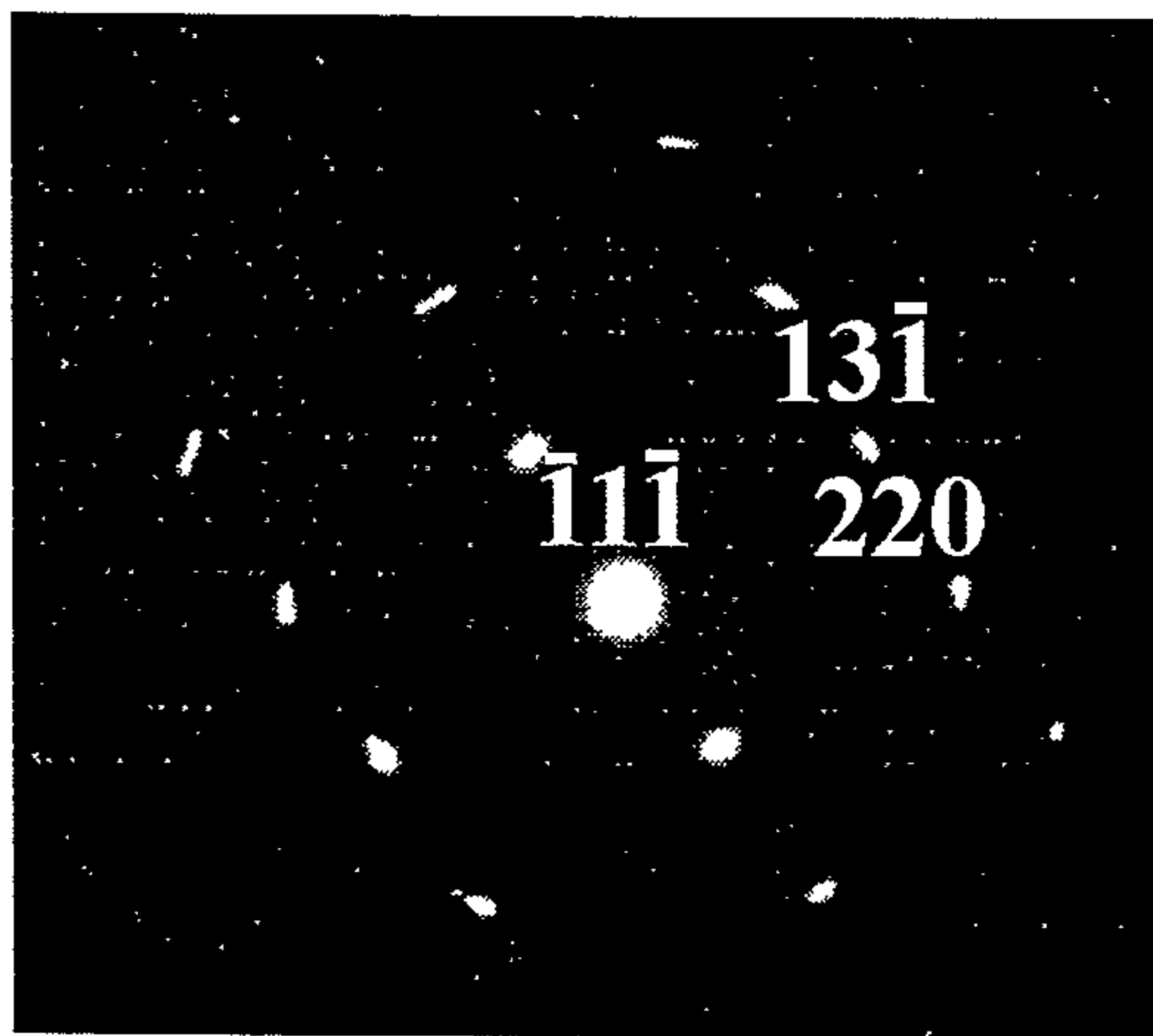


fig. 29

Fig.5.27 $[001]_{\text{fcc}}$ SAED pattern taken from one of these bands in fig.5.22 within the twinned structure.

Fig.5.28 $[011]_{\text{fcc}}$ SAED pattern taken from one of these bands in fig.5.22 within the twinned structure.

Fig.5.29 $[\bar{1}12]_{\text{fcc}}$ SAED pattern taken from one of these bands in fig.5.22 within the twinned structure.

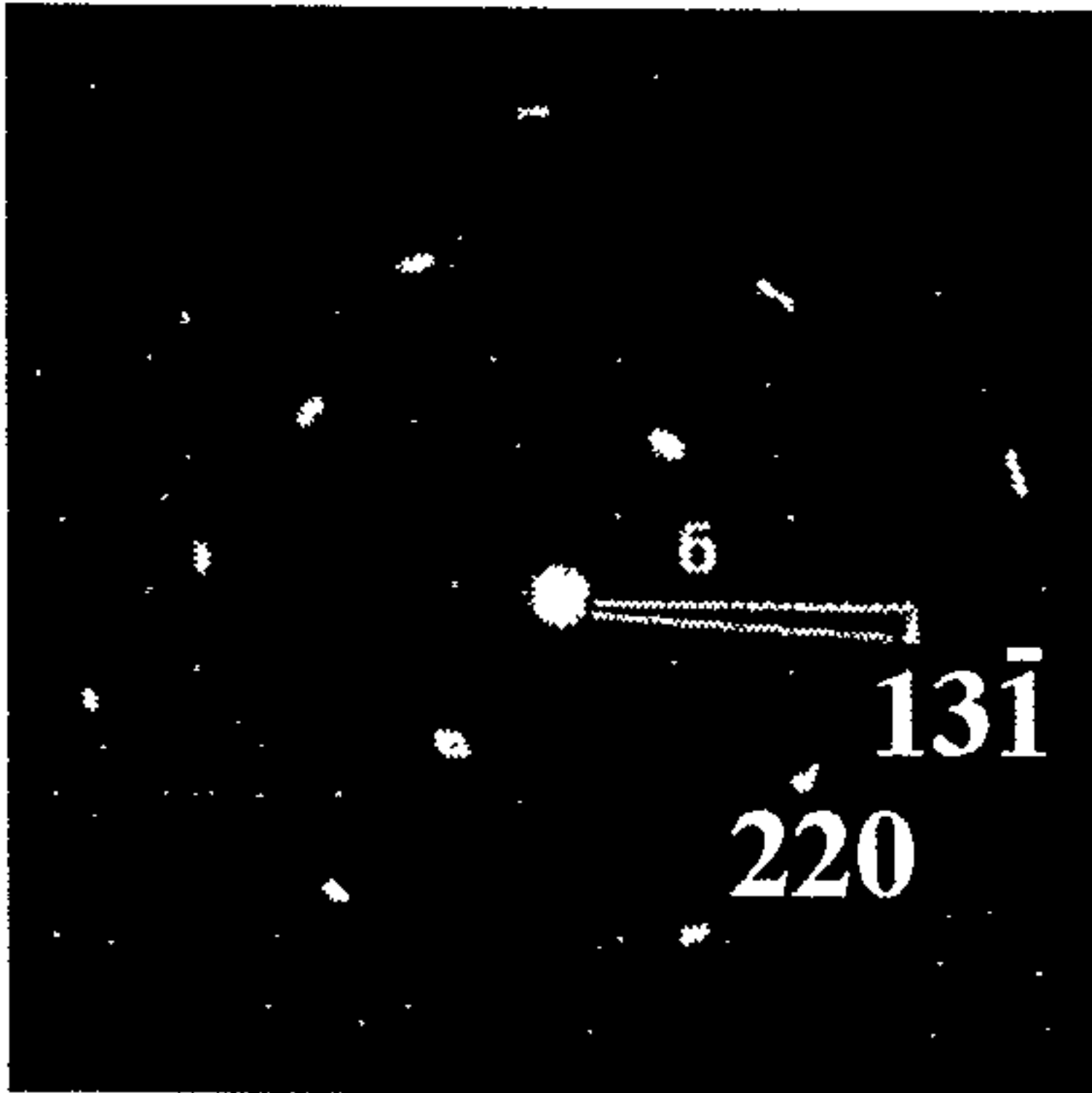


Fig. 5.30



fig. 5.31

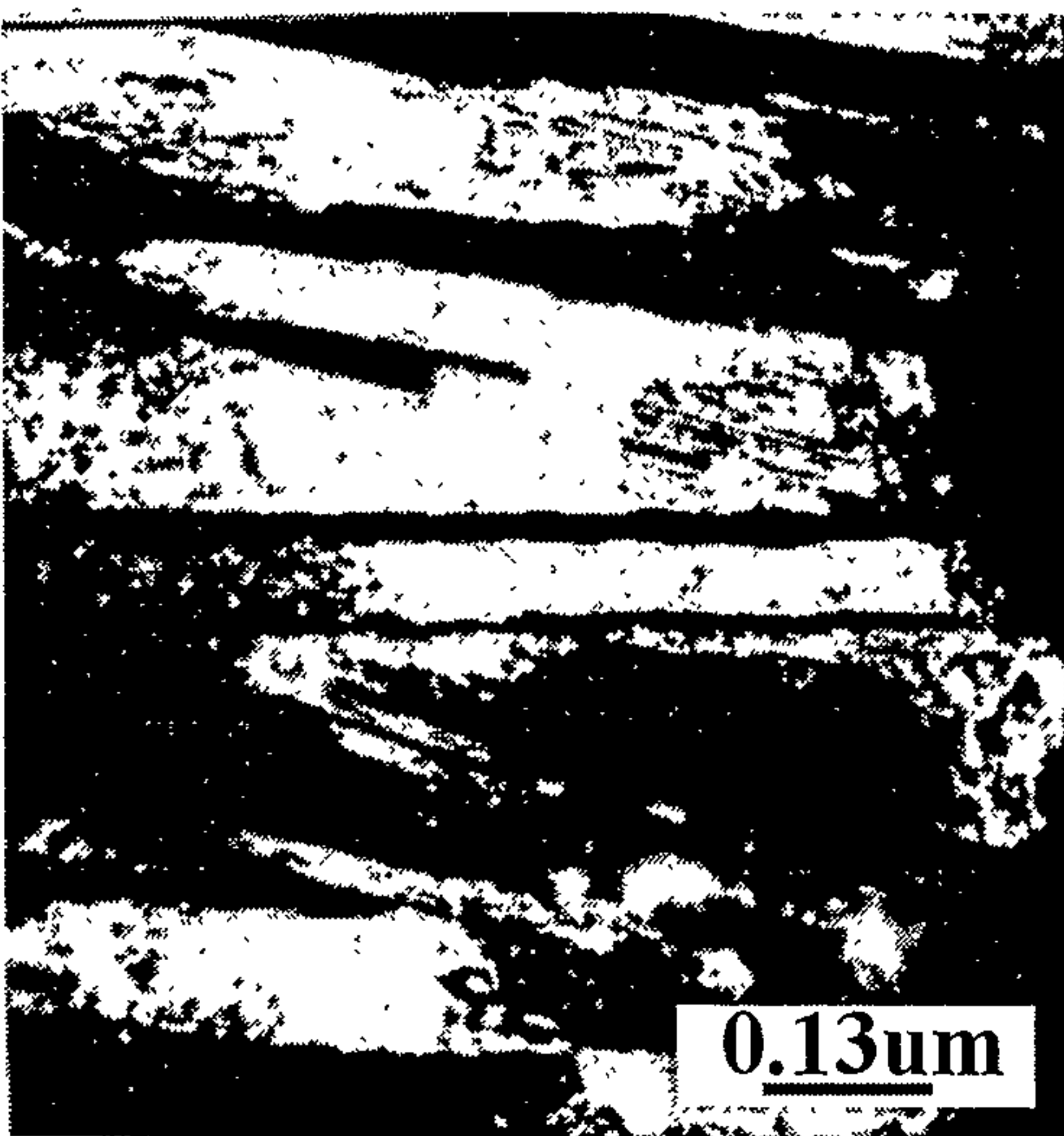


fig. 5.32

Fig.5.30 $[\bar{1}12]_{fcc}$ SAED pattern taken from the arrowed grain in the image of fig.5.31. The elongated diffraction spots are due to small angle twist boundaries between individual plate.

Fig.5.31 BF image taken from the longitudinal section of the WS wires. The angle between the arrowed bands is about 6° .

Fig.5.32 DF image which taken with the $(13\bar{1})$ reflection in fig.5.30. The angle between the arrowed bands is also about 6° .

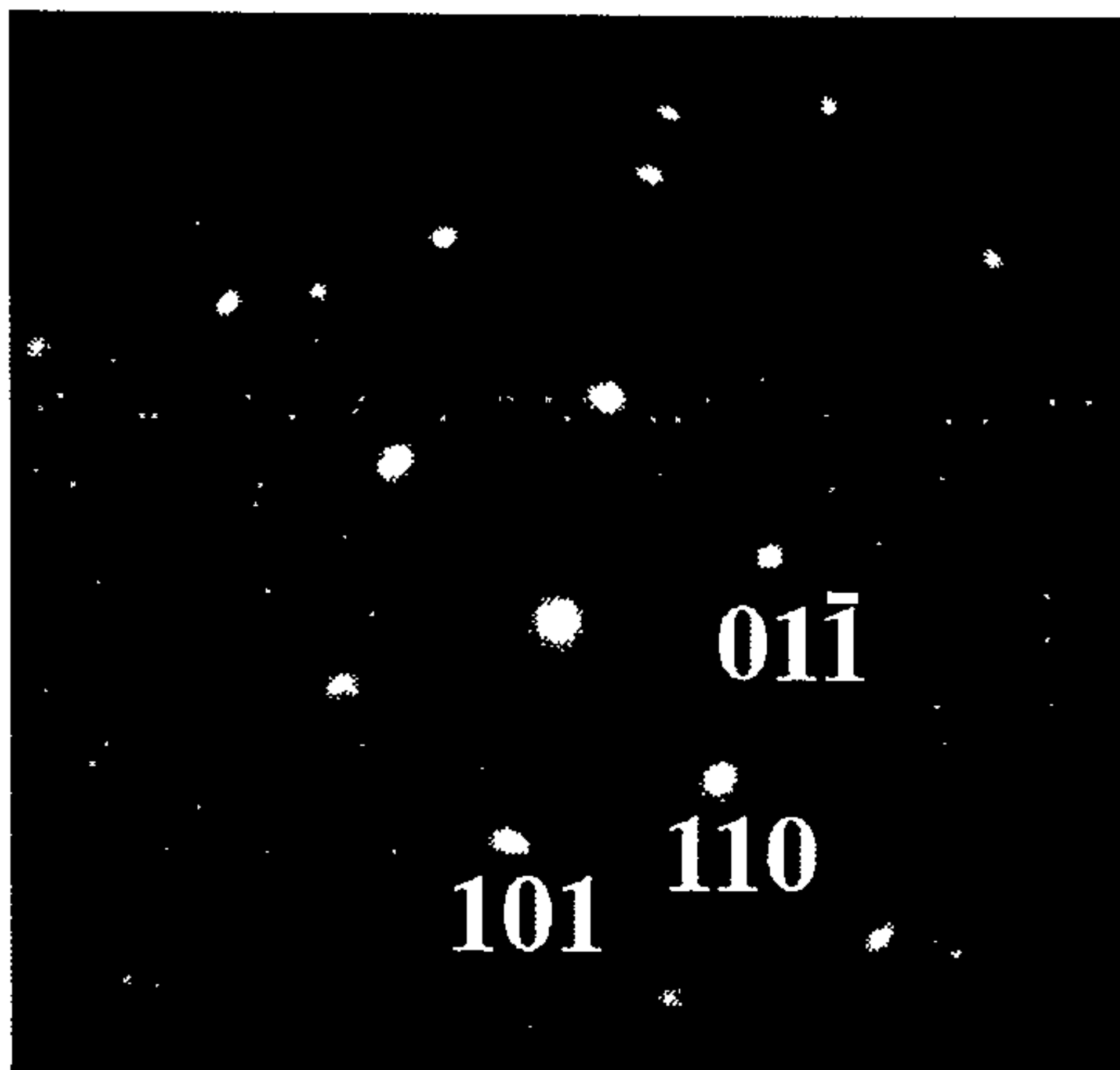


fig. 5.33

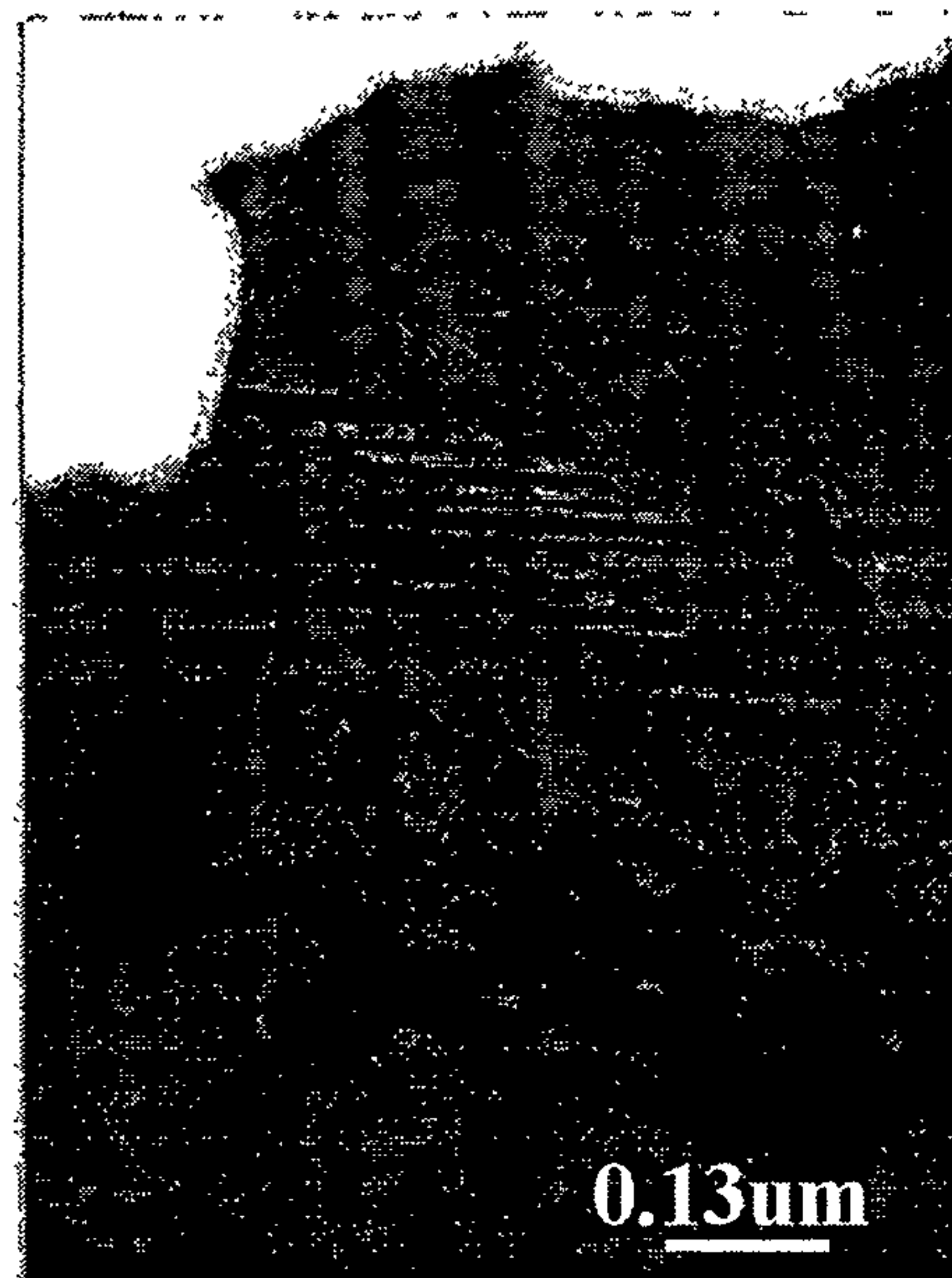


fig. 5.34



fig. 5.35

Fig.5.33 $[\bar{1}11]_{\text{bcc}}$ SAED pattern taken from the microtwinning plate in fig.5.34.

Fig.5.34 BF image taken from the longitudinal section of the WS wires. It shows the martensite phase plate in the WS wires is clearly recognised as band microtwinning structure.

Fig.5.35 DF image which taken by using the (110) spot in fig. 5.33. This DF image clearly revealing the microtwinning plate of the martensitic phase.

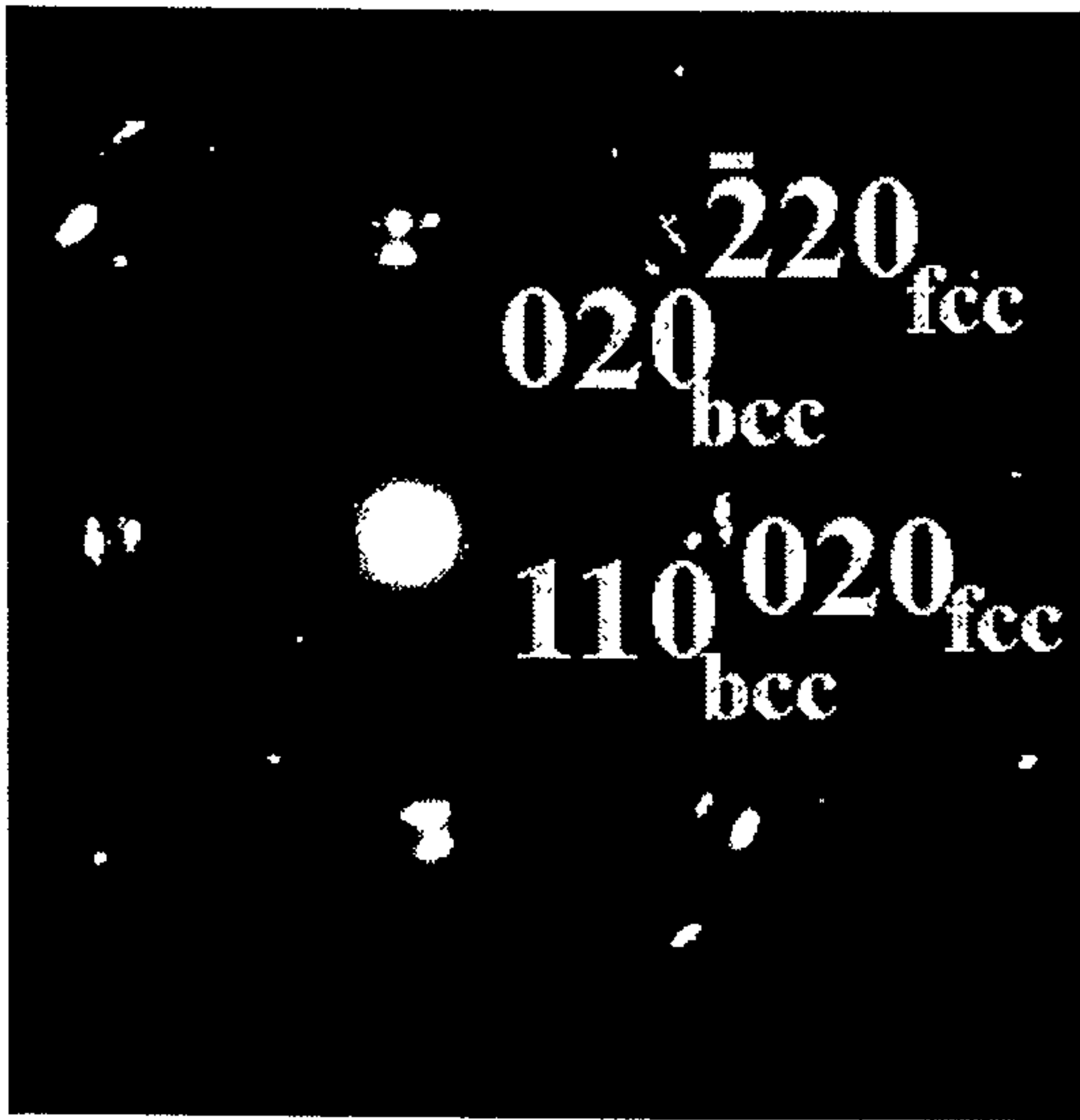


fig. 5.36



fig.5.37

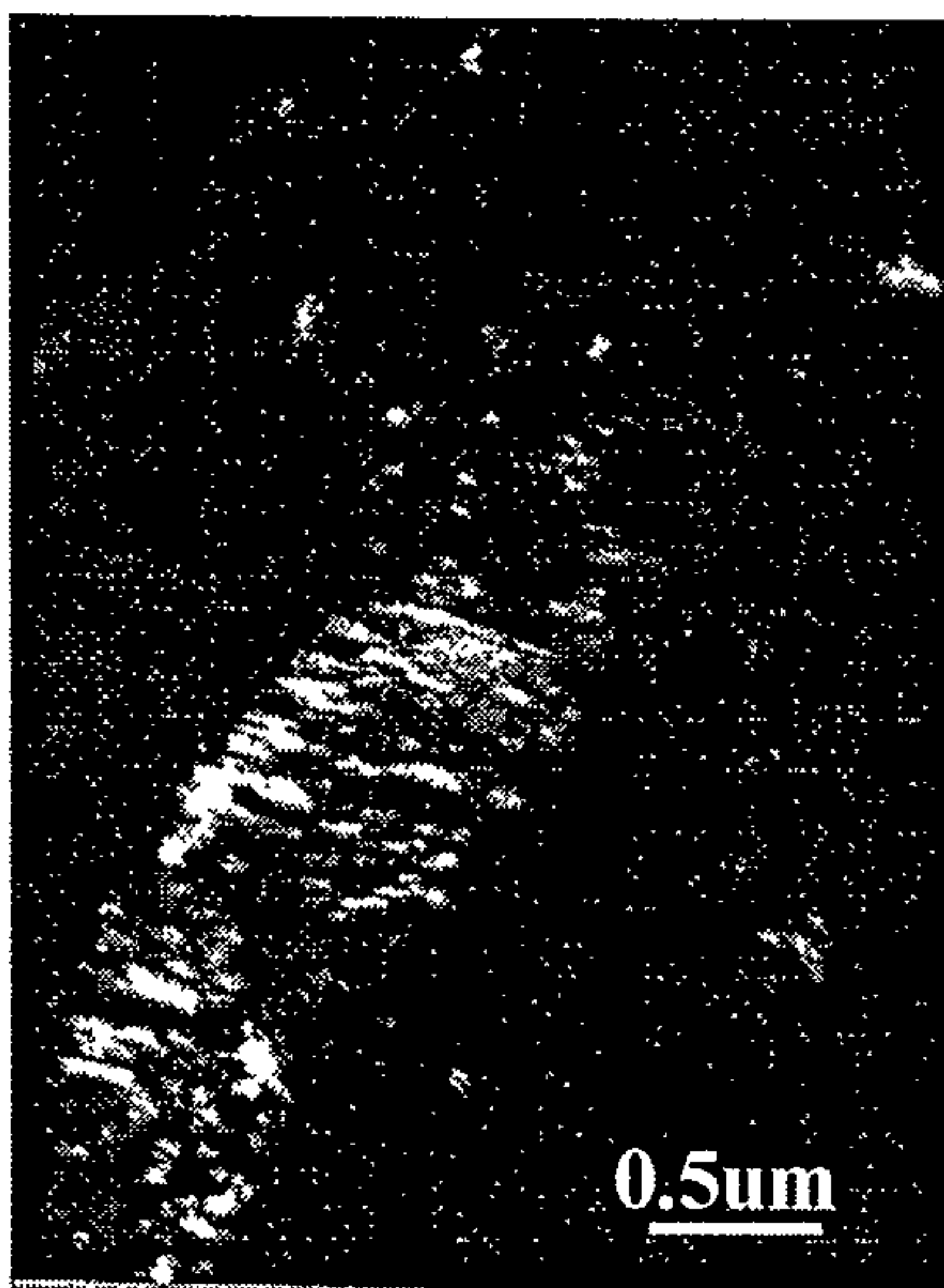


fig. 5.38

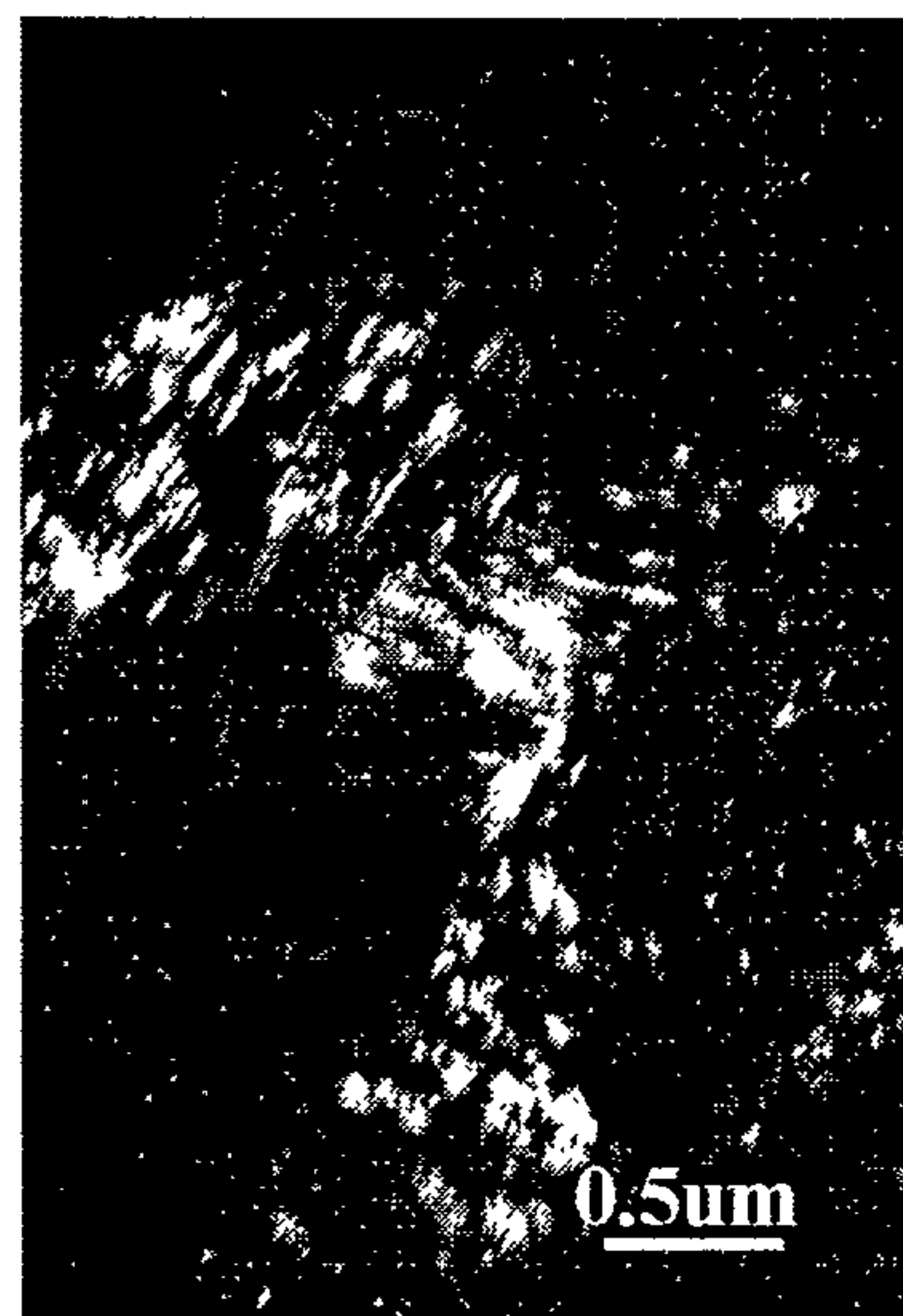


fig. 5.39

Fig.5.36 SAED pattern taken from the image of fig.5.37 showing $[001]_{fcc}$ pattern together with a $[001]_{bcc}$ pattern.

Fig.5.37 BF image taken from the cross-section of WS wires.

Fig.5.38 DF images taken using the martensite (110) spot in fig.5.36.

Fig.5.39 DF image taken using the austenite (020) spot in fig. 5.36.

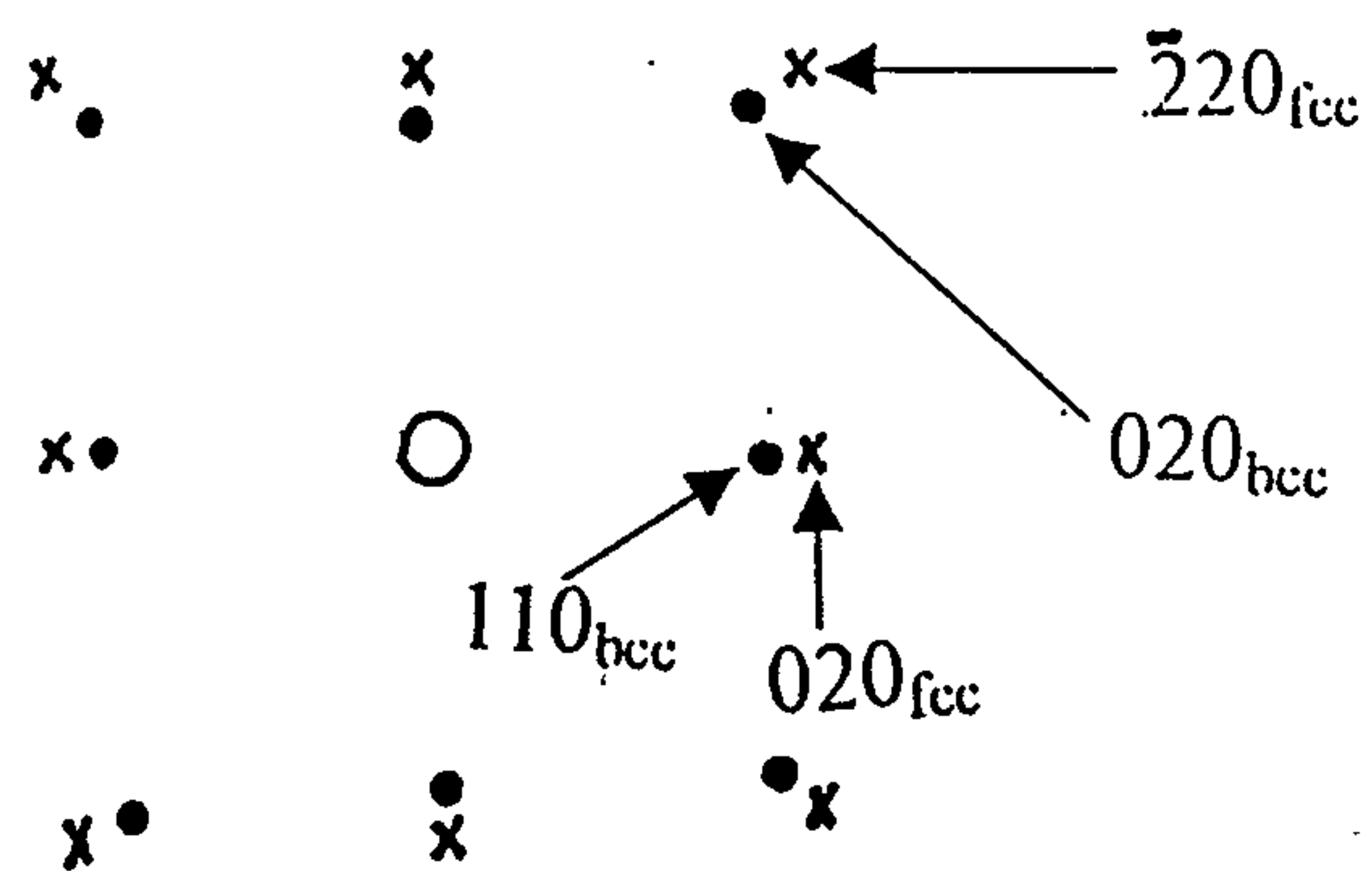


Fig.5.36' Diffraction pattern showing $[001]_{fcc}$ together with $[001]_{bcc}$.

$\times$ =austenitic phase; $\bullet$ =martensitic phase.

5.3.2.2.2. WF wires

A series of SAED patterns and BF images was taken to find the phases and morphology of the WF wires. Figs.5.40, 5.41, 5.42 and 5.43 are the $[011]_{\text{fcc}}$, $[001]_{\text{fcc}}$, $[112]_{\text{fcc}}$ and $[114]_{\text{fcc}}$ diffraction patterns taken with a convergent beam, from the longitudinal section WF wires (fig.5.3). The corresponding calculated lattice parameters are $3.65 \pm 0.03 \text{ \AA}$, $3.65 \pm 0.03 \text{ \AA}$, $3.68 \pm 0.02 \text{ \AA}$ and $3.64 \pm 0.04 \text{ \AA}$. These values are consistent with the austenitic phase lattice parameter ($3.68 \text{ \AA}$) and so indicate an austenitic structure.

The image in fig.5.44 was taken from the longitudinal section WF wires. It clearly shows twinning, but due to the difficulty of tilting such fine grains, no twin diffraction pattern could be taken to confirm it.

The martensite phases are confirmed by the selected area diffraction pattern (fig.5.45), which is taken from a small area in fig.5.44. This pattern is indexed as $[111]_{\text{bcc}}$ with a lattice parameter of $2.90 \pm 0.03 \text{ \AA}$. This value is consistent with the martensite lattice parameter. This indicates that martensite phases are present in the final Wilcock's wires. The elongated reflection spots in fig.5.45 are due to the presence of small angle twist boundaries.

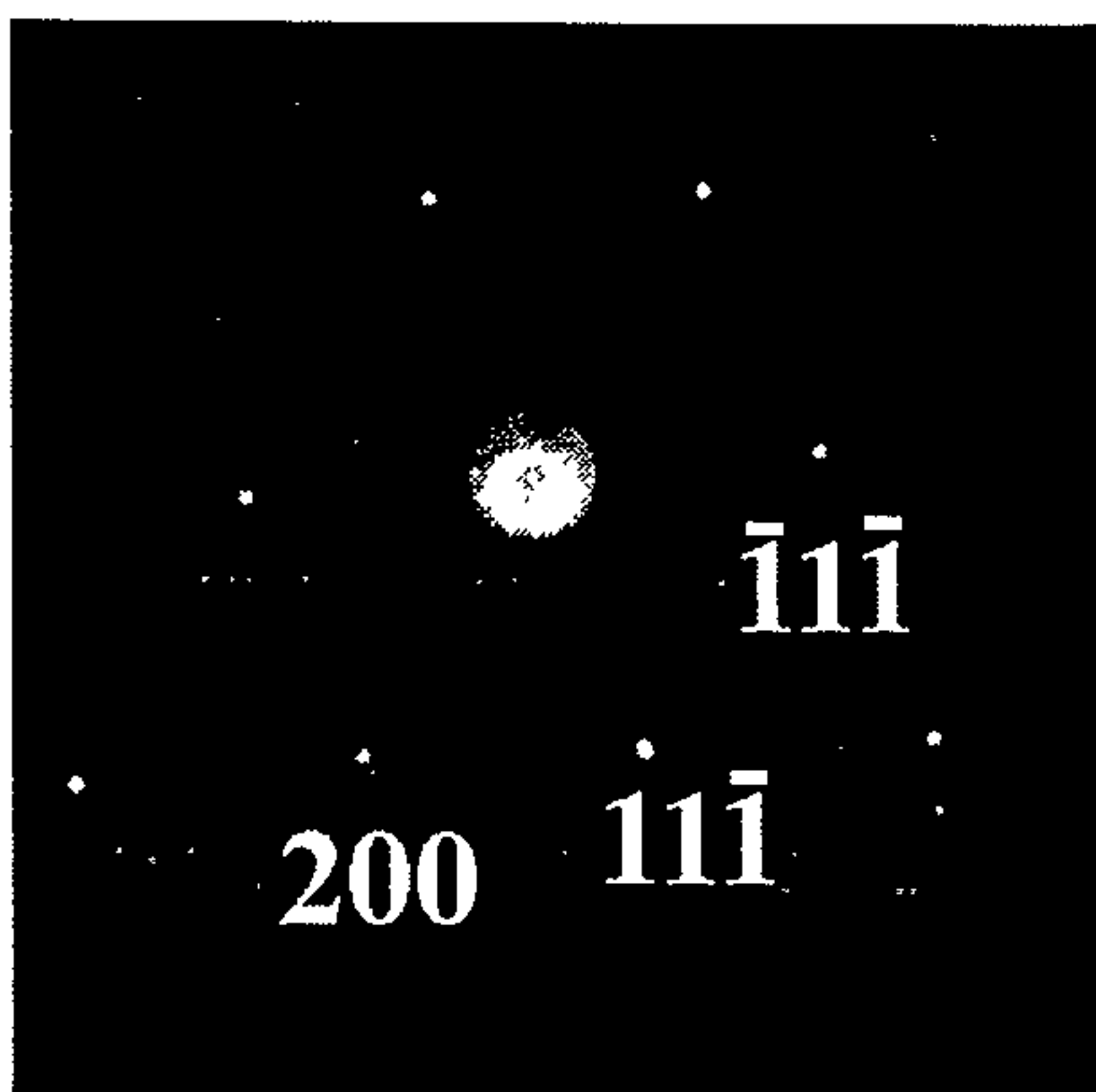


Fig. 5.40

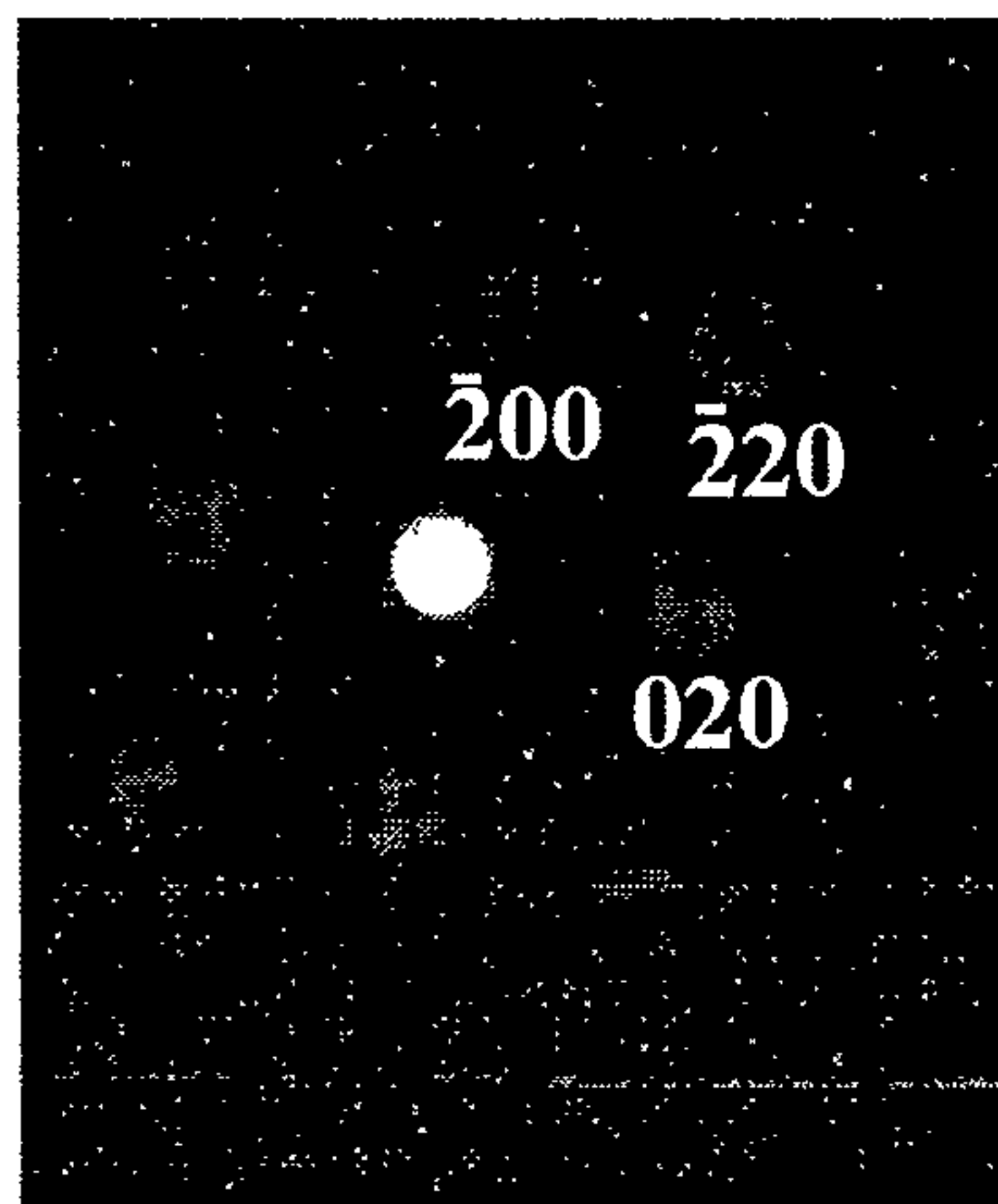


fig. 5.41

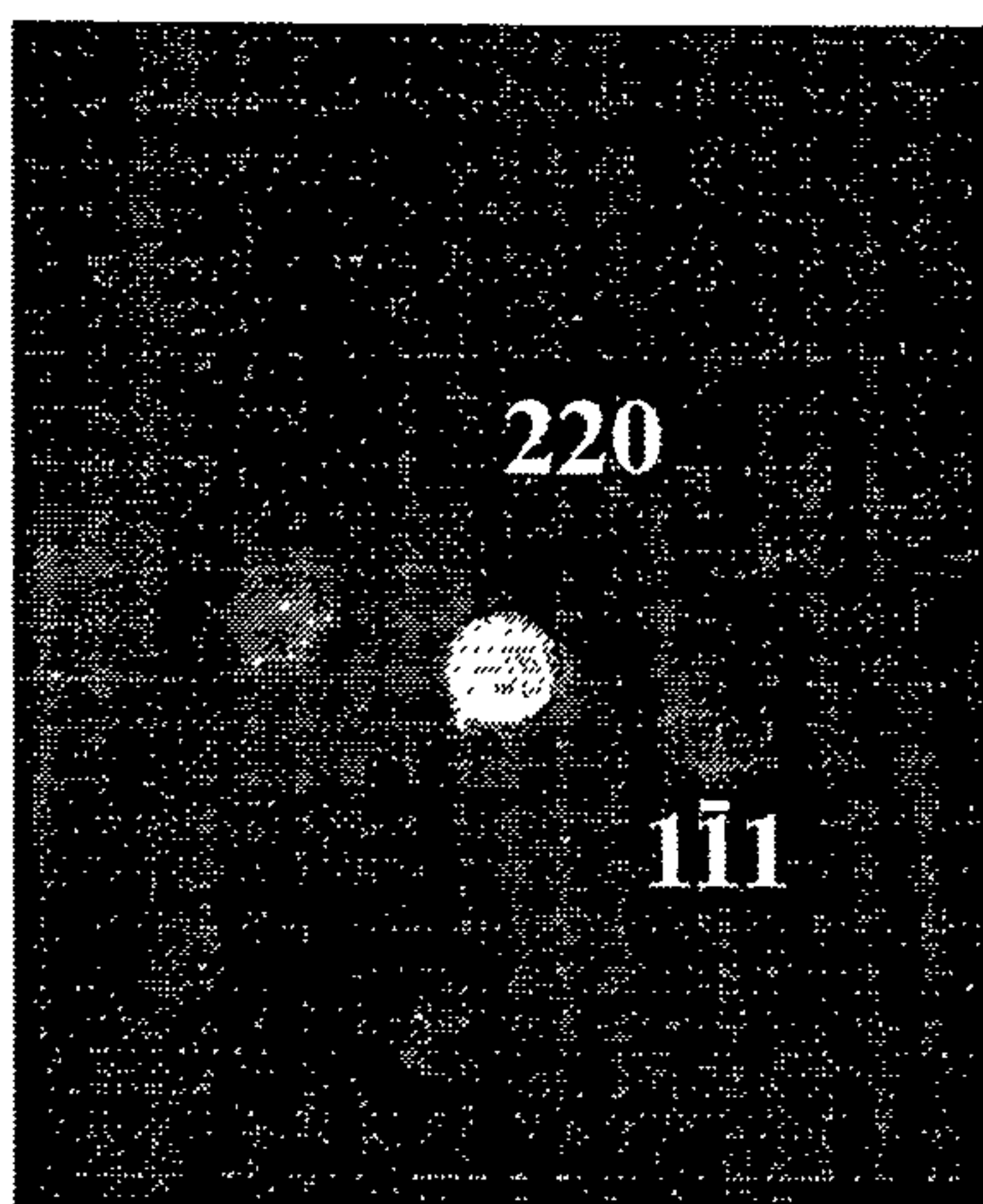


fig. 5.42

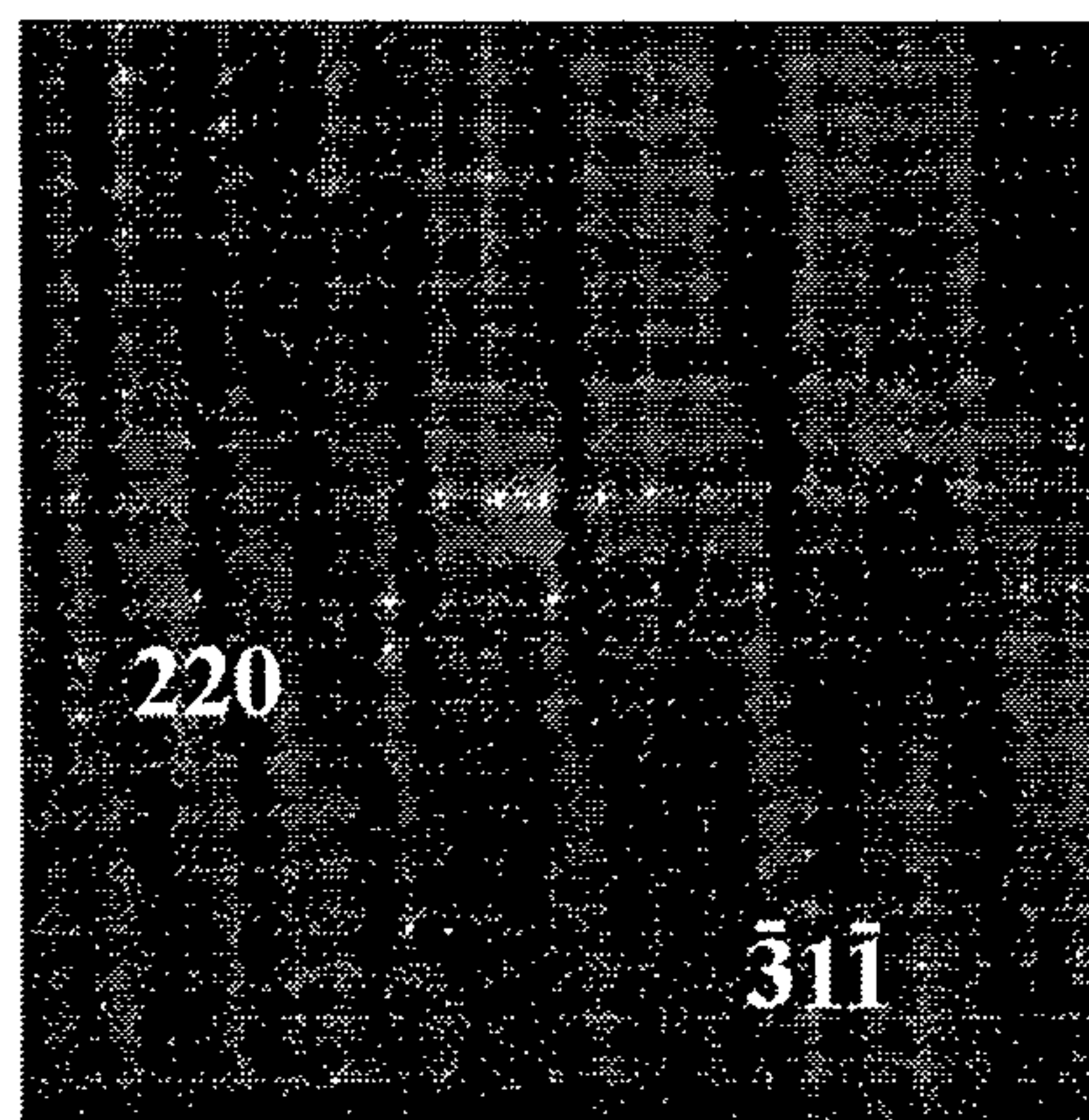


fig. 5.43

Fig.5.40 $[011]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section WF wire.

Fig.5.41 $[001]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section WF wire.

Fig.5.42 $[\bar{1}12]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section WF wire.

Fig.5.43 $[\bar{1}14]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section WF wire.

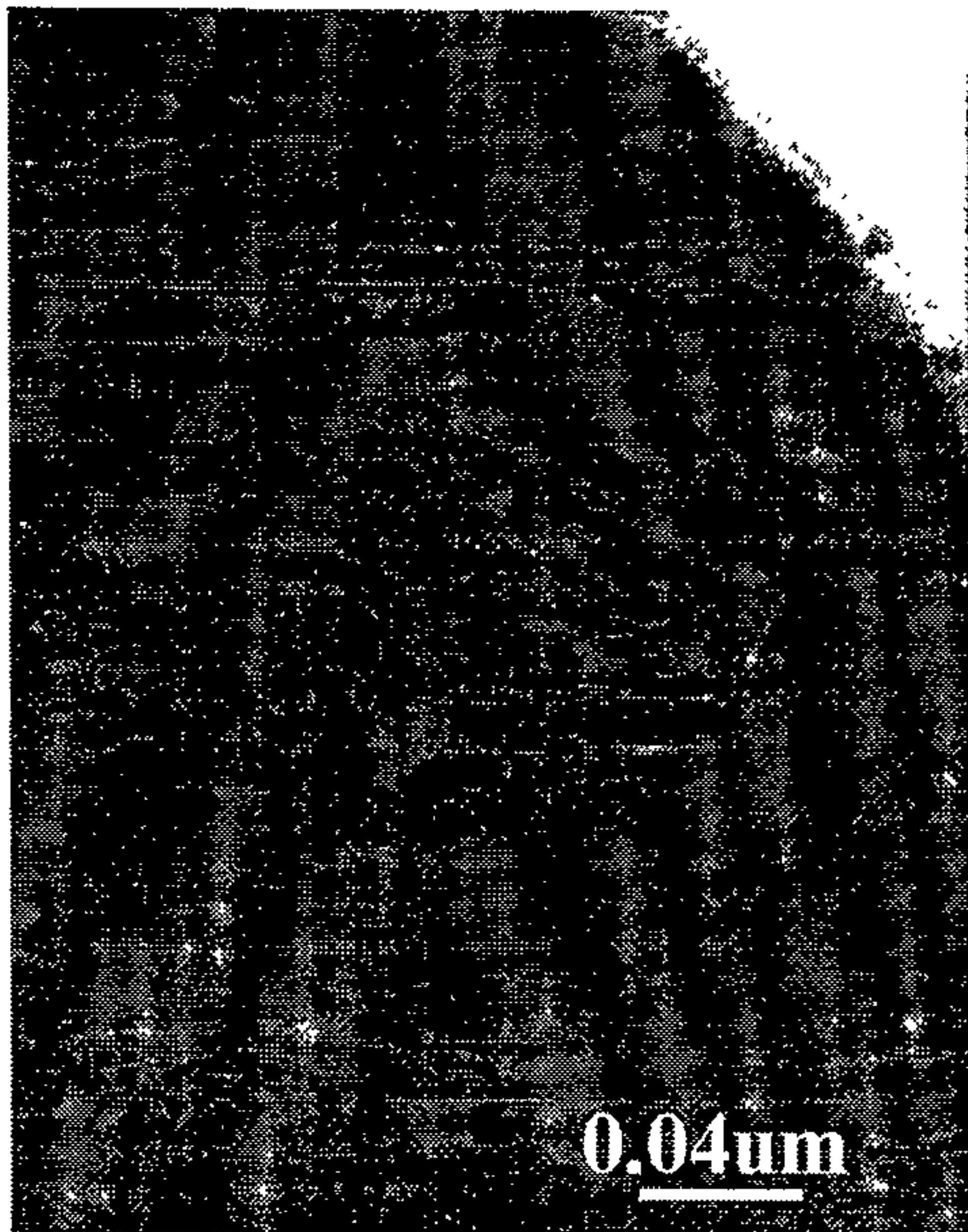


fig. 5.44

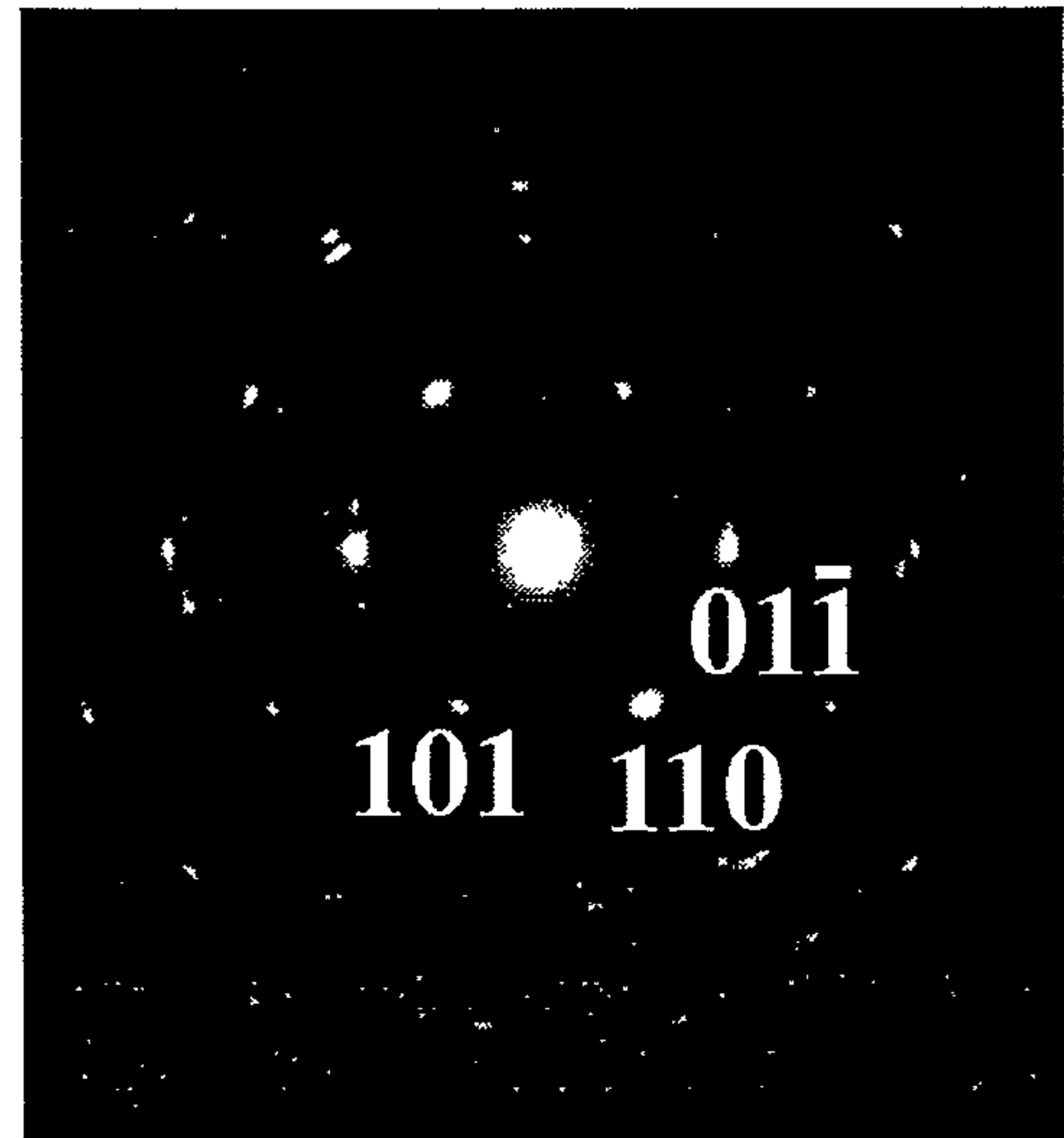


fig. 5.45

Fig.5.44 BF image taken from the longitudinal section WF wire. It clearly revealing martensitic phase is the band twinning structure.

Fig.5.45 $[\bar{1}11]_{bcc}$ SAED pattern taken from the longitudinal section WF wire.

5.3.2.2.3. GS wires

A series of SAED patterns, BF and DF image were taken to find the phases and morphology of the GS wires. Figs.5.46, 5.47, 5.48, 5.49 & 5.50 are the diffraction patterns taken from the longitudinal and cross section GS wires (figs.5.5, & 5.14). They can be indexed as $[001]_{fcc}$, $[112]_{fcc}$, $[233]_{fcc}$, $[011]_{fcc}$ and $[111]_{fcc}$. The corresponding calculated lattice parameters are $3.67 \pm 0.03 \text{ \AA}$, $3.67 \pm 0.03 \text{ \AA}$, $3.63 \pm 0.03 \text{ \AA}$, $3.66 \pm 0.03 \text{ \AA}$ and $3.69 \pm 0.04 \text{ \AA}$. These values are consistent with the austenitic phase lattice parameter ($3.68 \text{ \AA}$) and indicate an austenitic structure.

The stripe shown in the BF imaged (fig.5.52) has been identified as an austenitic twin structure by the selected area diffraction pattern (fig.5.51).

The diffraction pattern in fig.5.51 can be separated into two: $[101]_{fcc}$ and $[101]_{fcc}$ as shown in fig.5.51'. The calculated lattice parameters for both patterns are $3.62 \pm 0.04 \text{ \AA}$. This value is consistent with an austenitic phase lattice of $3.65 \text{ \AA}$. Thus, it indicated this stripe is an austenitic twinning structure. The austenitic twin zone axis is $[101]$ and the twin plane is (111) . Fig. 5.53 is the dark field image obtained using $(111)\gamma+t$ diffraction spots in fig.5.51. This dark field image further confirms that this stripe is due to the twin austenitic structure.

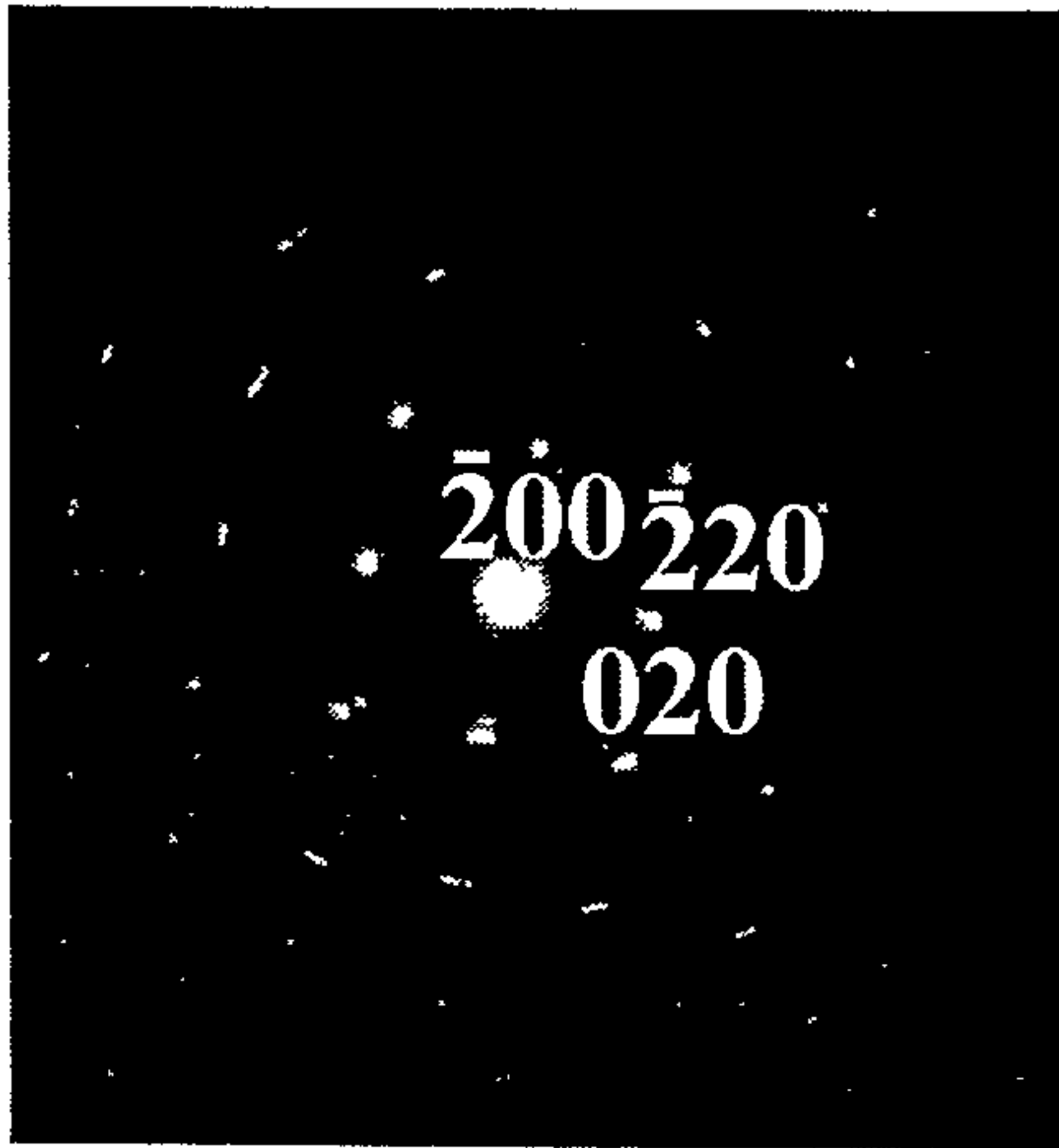


fig. 5.46

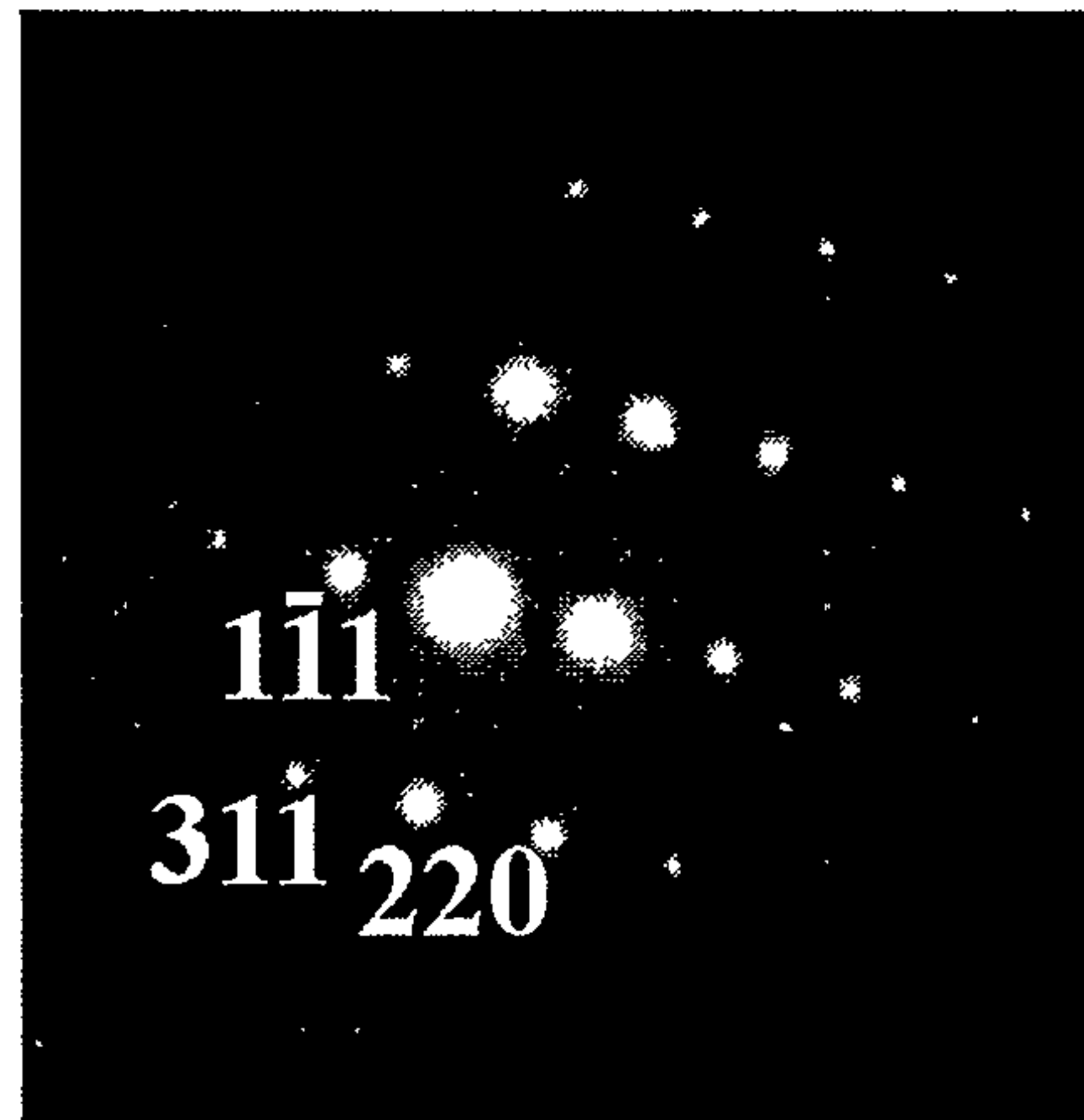


fig. 5.47

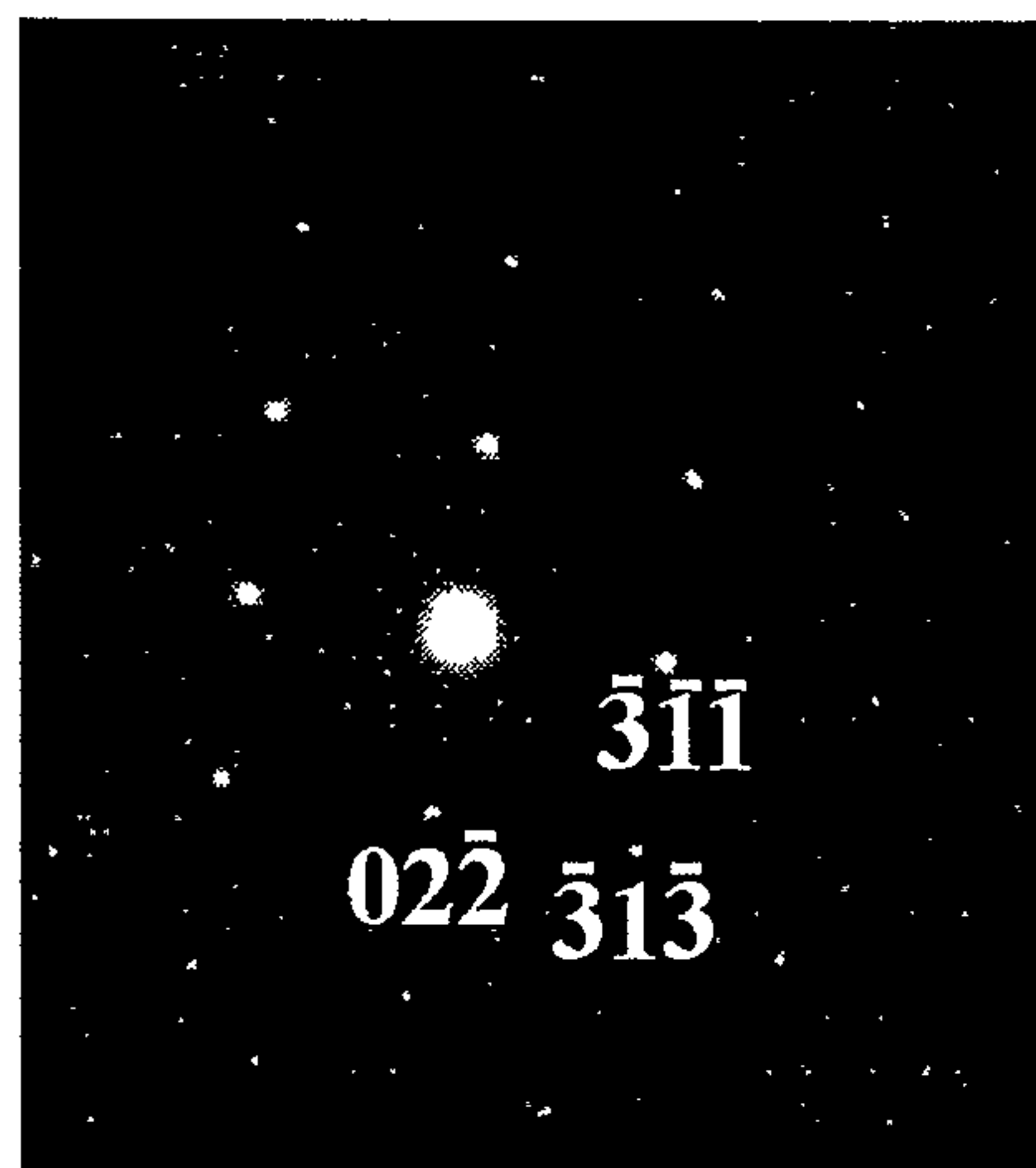


fig. 5.48

Fig.5.46 $[001]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section GS wire.

Fig.5.47 $[112]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section GS wire.

Fig.5.48 $[233]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section GS wire.

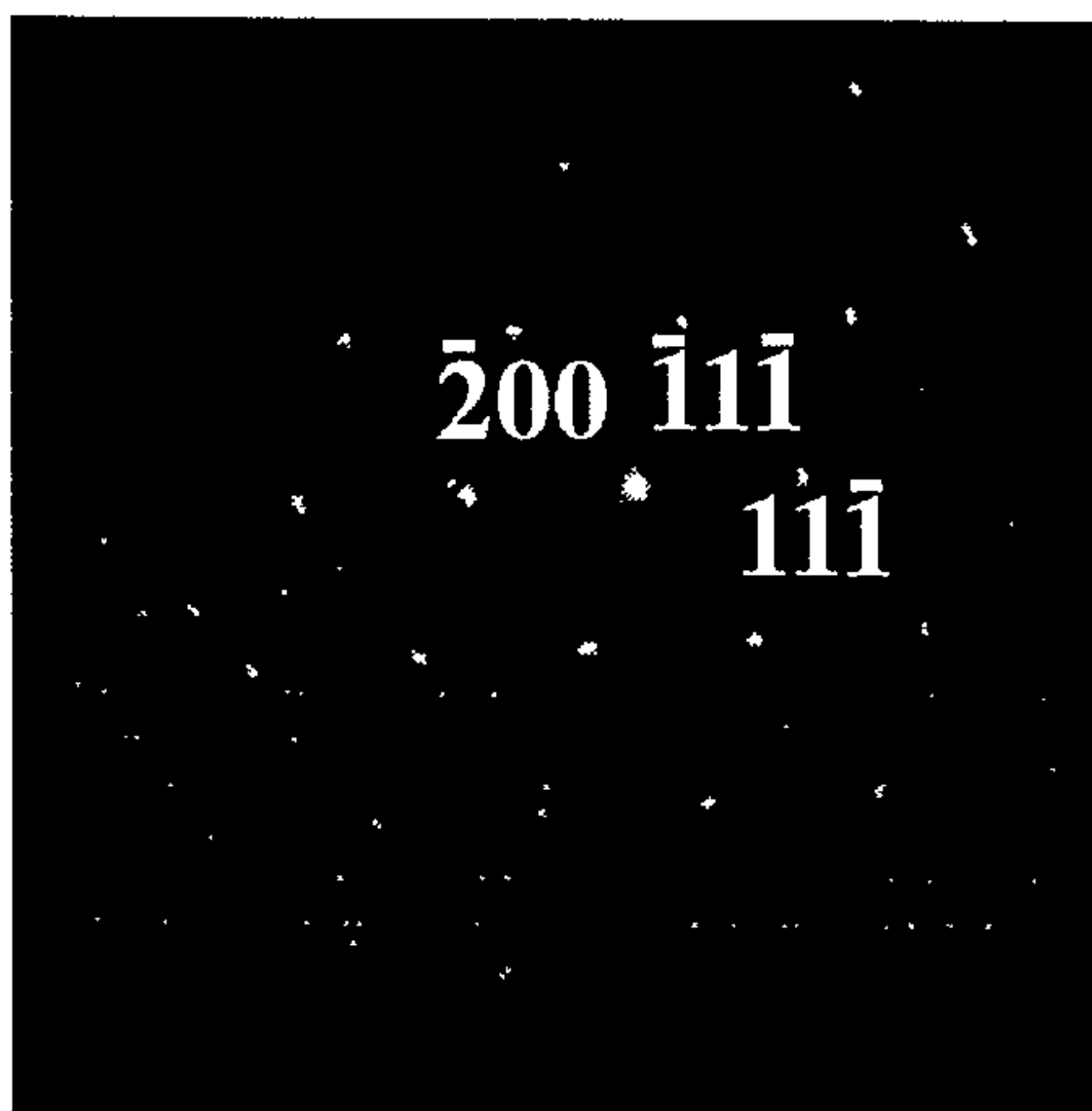


fig. 5.49

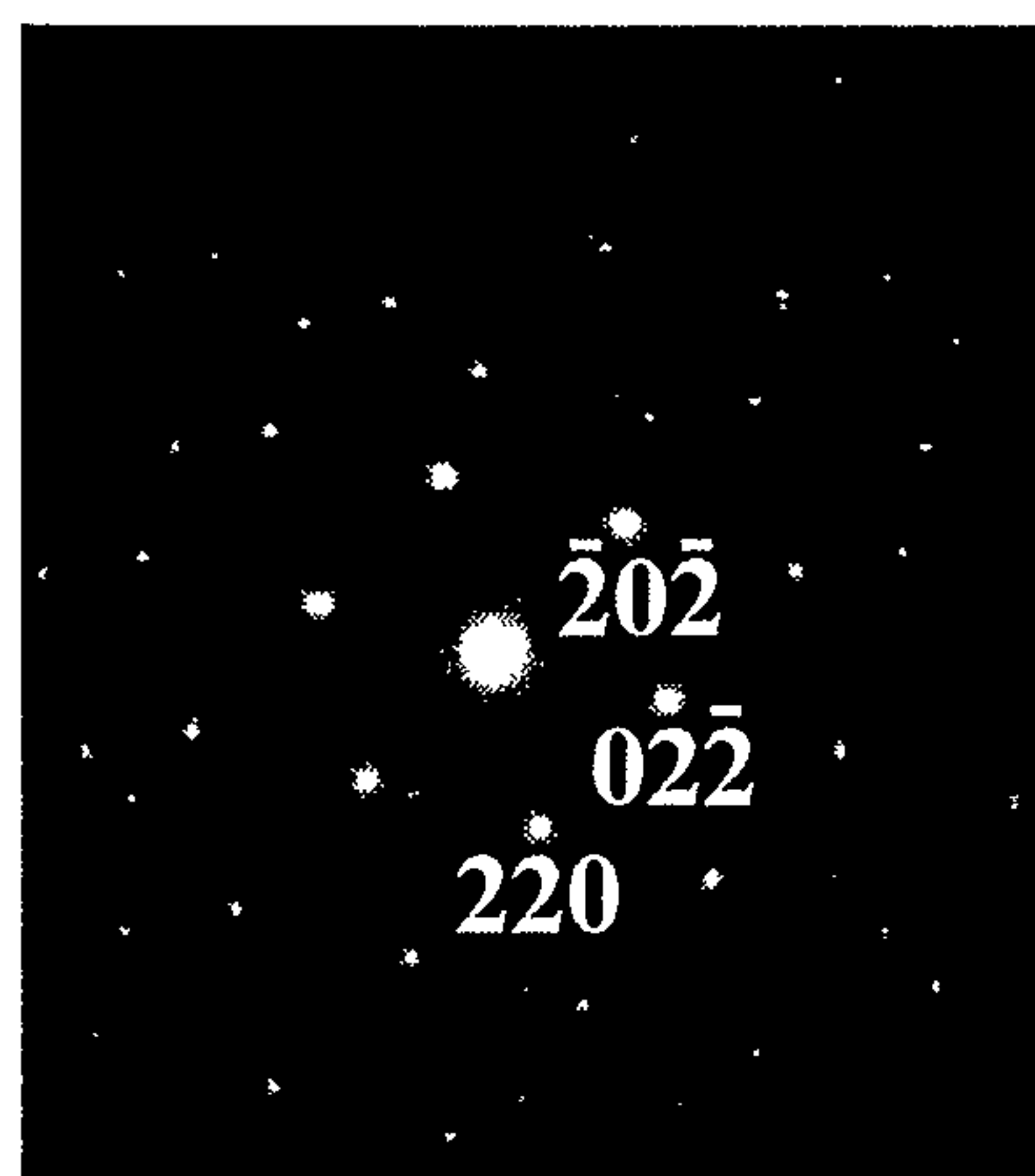


fig. 5.50

Fig.5.49 $[011]_{\text{fcc}}$ SAED pattern taken from the relative cross section GS wire.

Fig.5.50 $[\bar{1}11]_{\text{fcc}}$ SAED pattern taken from the relative cross section GS wire.

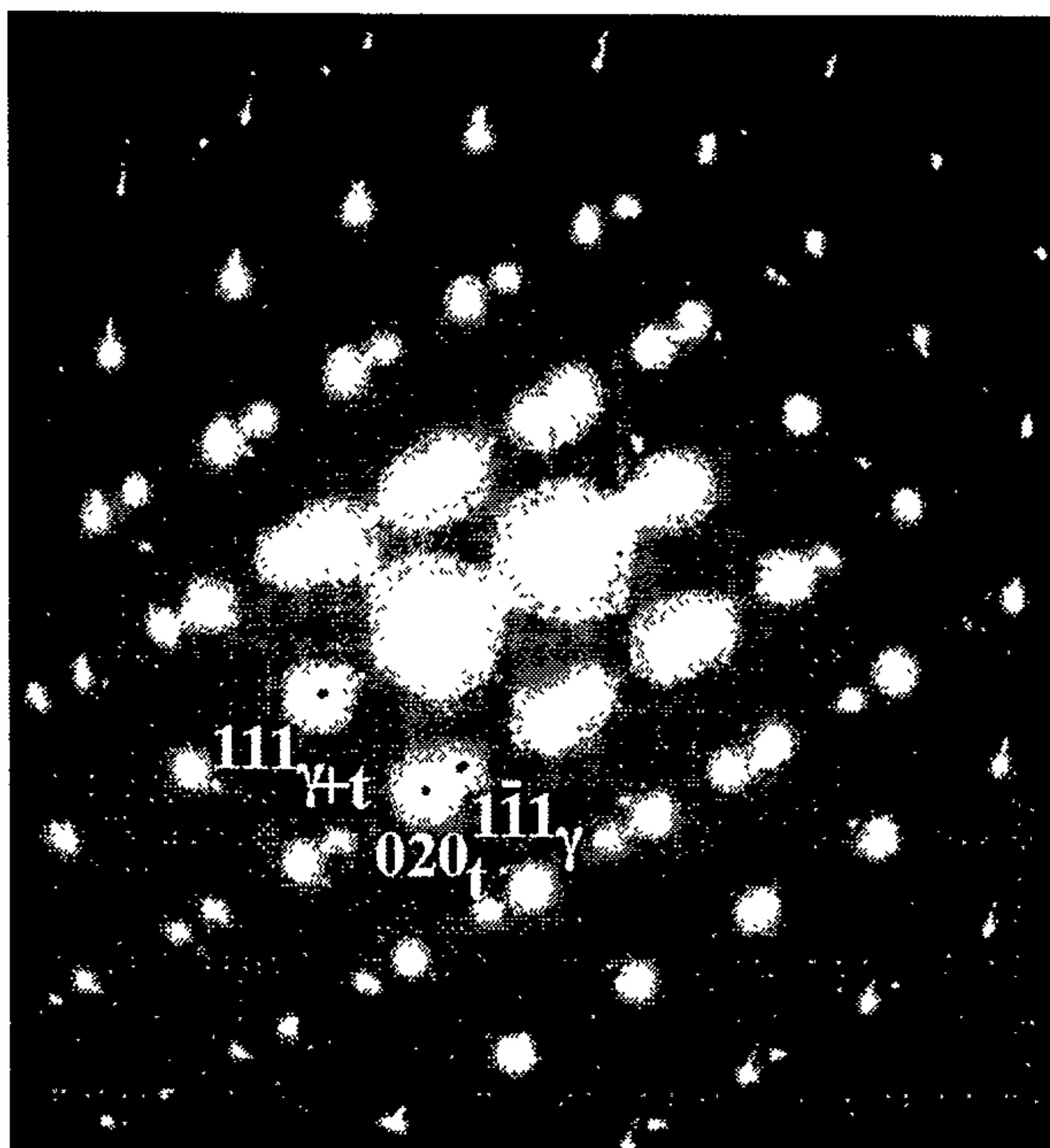


fig. 5.51



fig. 5.52



fig. 5.53

Fig.5.51 SAED pattern taken from the stripe shown in fig.5.52, and clearly revealing austenite twinning structure.

Fig.5.52 BF image taken from the cross section GS wire.

Fig.5.53 DF image obtained using $(111)_{\gamma+t}$ diffraction spots in fig.5.51. This dark field image confirms that the stripe in one of these grains in fig.5.51 is due to the twin austenitic structure.

5.3.2.2.4. GF wires

A series of SAED patterns, BF and DF images were taken to find the present phase and morphology of the GF wires. Fig.5.54 and fig.5.55 were indexed as the $[011]_{\text{fcc}}$ and $[112]_{\text{fcc}}$ diffraction patterns taken from the relative longitudinal section GF wires. The corresponding calculated lattice parameters are $3.66 \pm 0.02 \text{ \AA}$ and $3.62 \pm 0.05 \text{ \AA}$. These values are consistent with the austenitic phase lattice parameter ($3.68 \text{ \AA}$) and indicate an austenitic structure.

The martensite phase was confirmed by the $[111]_{\text{bcc}}$ martensite diffraction pattern (fig.5.56) which is taken from the select area in fig.5.6. The calculated lattice parameter is $2.85 \pm 0.03 \text{ \AA}$. This value corresponds to the martensite lattice parameter. This indicates that martensite is present in the GF wires.

The austenitic twinning structure has been positively identified (fig.5.57) in the GF wires. The diffraction pattern in fig.5.57 can be separated into two: $[101]_{\text{fcc}}$ and $[101]_{\text{fcc}}$ as shown in fig.5.57'. The calculated lattice parameter for both patterns is $3.62 \pm 0.05 \text{ \AA}$. This value is consistent with an austenitic phase lattice of $3.65 \text{ \AA}$. Thus, we can conclude that the diffraction pattern in fig.5.57 shows this band structure to be an austenitic twinning structure. The austenitic twin zone axis is $[101]$ and twin plane is (111) . Fig.5.58 is the dark field image using the spot (222) in fig 5.57. The elongated diffraction spots (222) are due to small angle twist boundaries between individual twin plates. The angle of (222) reflection spot in the diffraction pattern is about 10° . This can confirmed by observing the dark field image in fig.5.58. The

angle we measured between these bands in dark field image is about 10° also.

In summary, the use of TEM showed that both austenitic and martensitic phases were present in the WS, WF, and GF wires, whereas, only the austenitic phase was found in the GS wires. Both austenite and martensite phases have the similar band twinning microstructure morphology in the WS, WF and GF wires. Small angle twist boundaries exist in the WS, WF and GF wires. Austenite twinning exists in WS, GS and GF wires and probably in the WF wires as well.

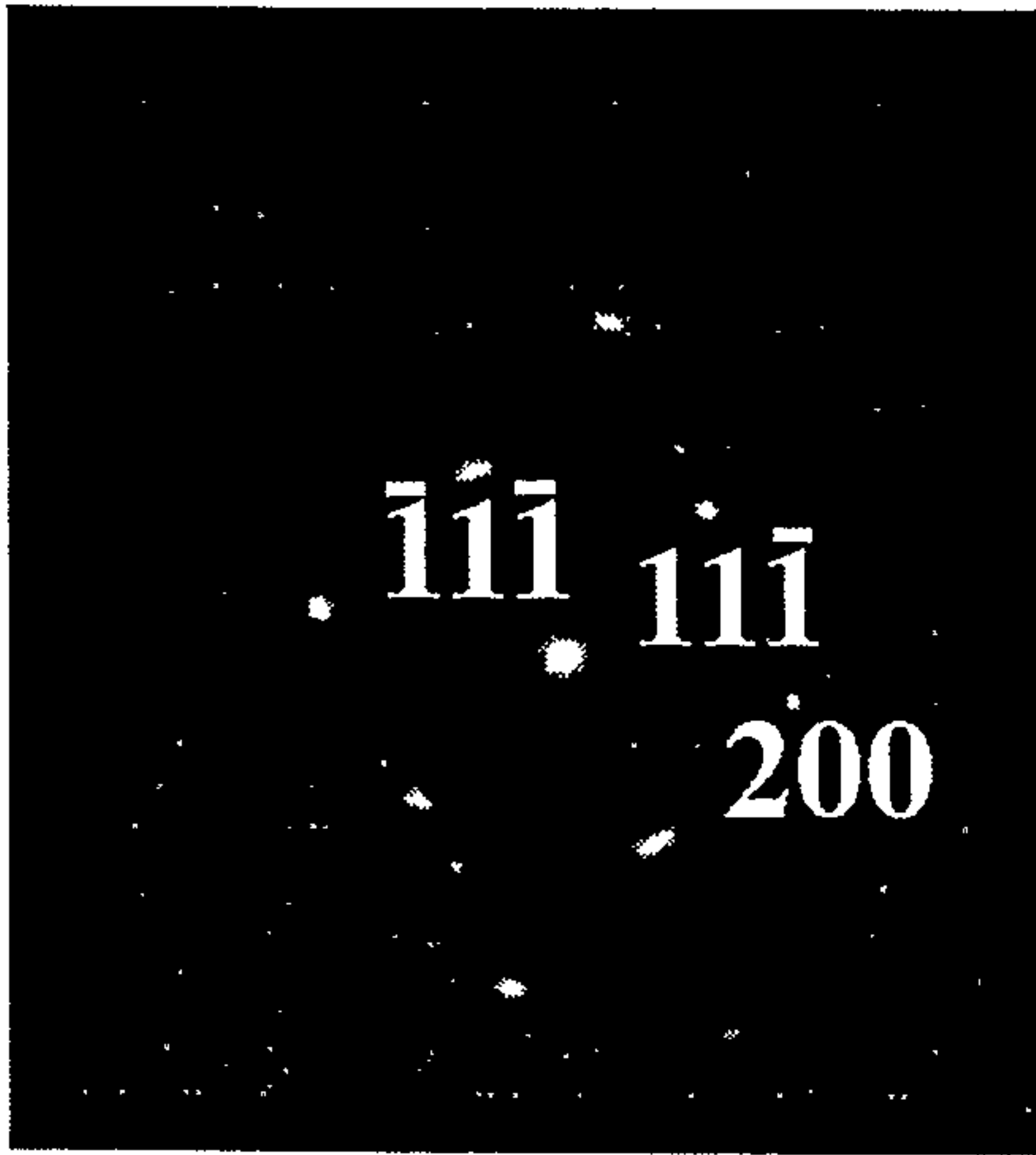


fig. 5.54



fig. 5.55

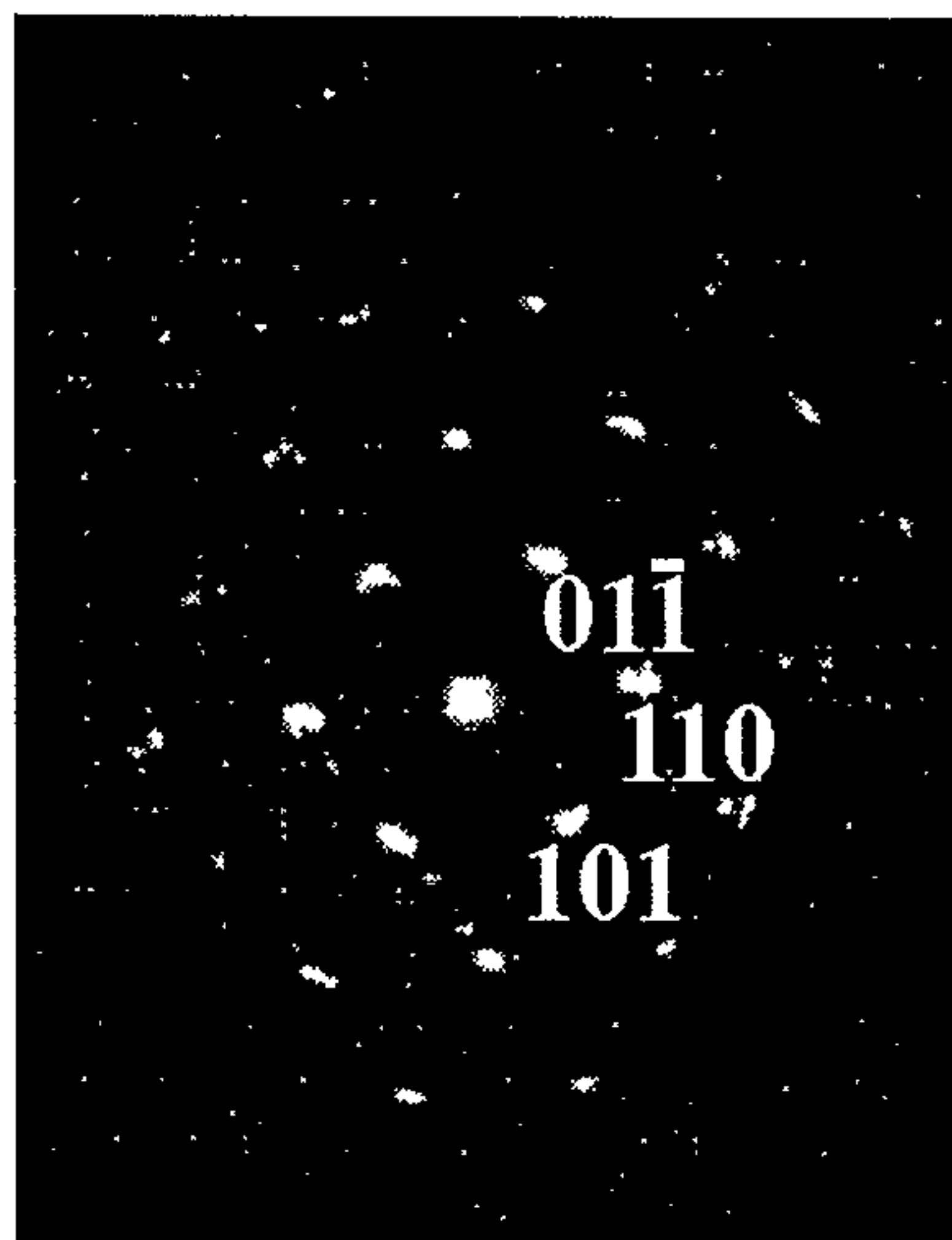


fig. 5.56

Fig.5.54 $[011]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section GF wire.

Fig.5.55 $[\bar{1}12]_{\text{fcc}}$ SAED pattern taken from the relative longitudinal section GF wire.

Fig.5.56 $[\bar{1}11]_{\text{bcc}}$ SAED pattern taken from the longitudinal section GF wire.

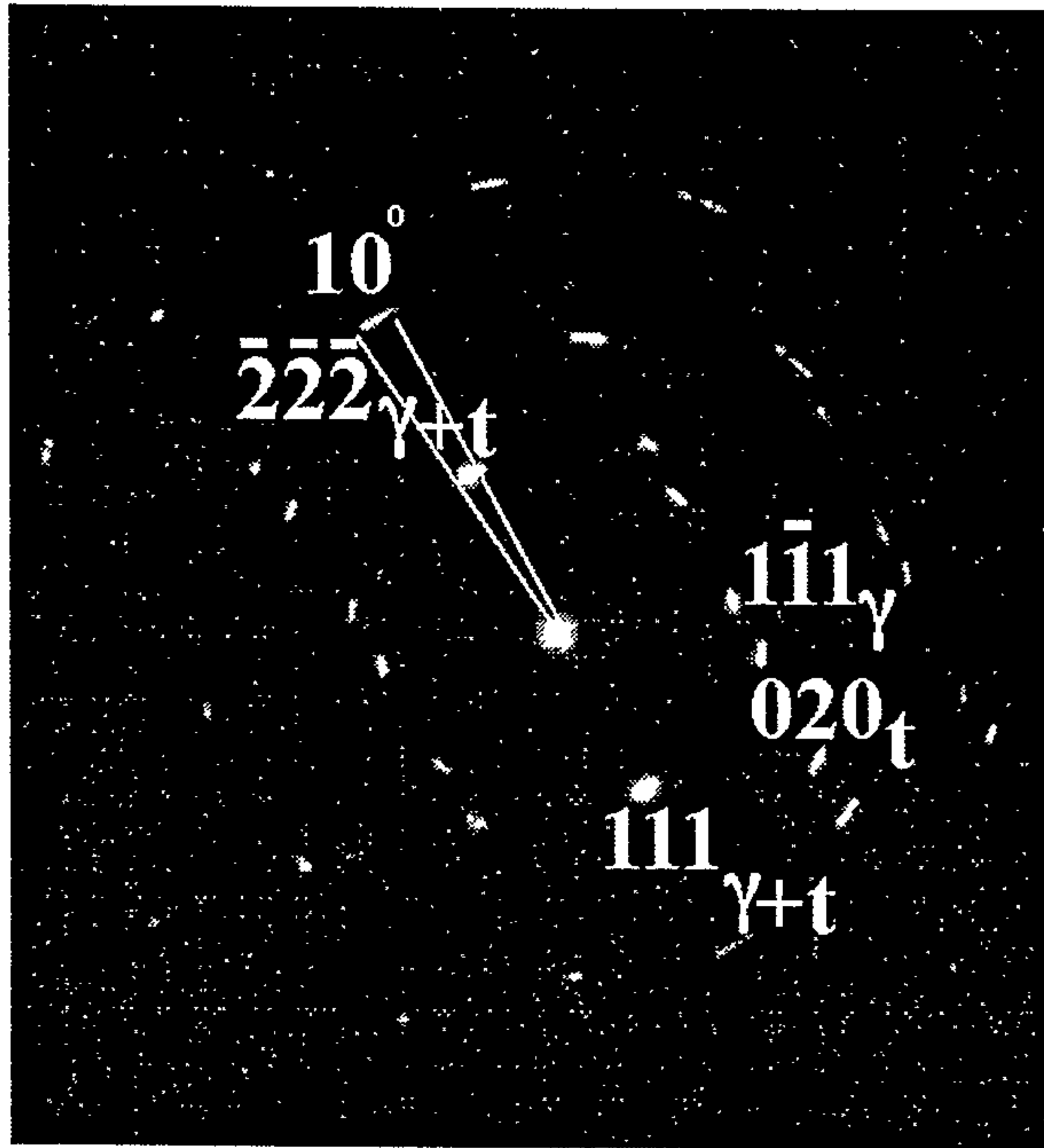


fig. 5.57



fig. 5.58

Fig.5.57 SAED pattern taken from the area show in the fig.5.58 and clearly revealing twinning. The elongated diffraction spots are due to small angle twist boundaries between individual plate.

Fig 5.58 DF image using the spot $(\bar{2}\bar{2}\bar{2})_{\gamma+t}$ in fig 5.57. It clearly showing the image of small angle twist boundaries between individual twin plates.

5.4. Discussion:

5.4.1. Grain size and grain shape:

The TEM results show that the order of the grain size from largest to smallest is

$$GS > WS > GF > WF.$$

This order is precisely the opposite of the ordering of the tensile 0.1% yield strengths as found in the mechanical property tests (chapter 3). These results (ie $GS > WS > GF > WF$) are consistent with the Hall-Petch equation which expresses the relationship between strength and grain size:[5.11, 5.30]

$$\sigma_Y = \sigma_0 + K_Y D^{-1/2}$$

σ_Y is yield strength obtained from the tensile test,

σ_0 is the friction stress opposing motion of dislocation,

K_Y is the "unpinning constant," measuring the extent to which dislocations are piled up at barriers,

D is average width of elongated grain diameter obtained from the TEM investigation.

The values of σ_Y and D for four wires are listed in table 5.4. These values in table 5.4 are plotted in fig.5F and equation of Hall-Petch is fitted to them by least squares. The values of σ_0 & K_Y obtained for this fit are $\sigma_0 = 299 \pm 30$ and $K_Y = 34200 \pm 2500$, where the errors are the S.D. of the least squares fitting.

The values of σ_0 & K_Y can be compared with the values given by Petch[38]

and discussed by Honeycombe[38]. The data from Petch shows that σ_0 lies in the range 80-140MNm⁻² and that K_Y is about 34000. (this value is obtained by changing the unit of σ_Y from MNm⁻² into MPa, and change the unit of D from mm into A). From the results of Petch (as discussed by Honeycombe), the K_Y term “has been found to be sensitive to temperature, composition and strain rate”, and it is. On the other hand, “ σ_0 is particularly insensitive to the temperature and composition” [38] and therefore it is not surprising that it is different from the value obtained here (80-140 c.f. 299).

Table.5.4:

	$\sigma_{Y(Mpa)}$ Yield strength	D(A) Average grain size	$D^{-1/2}$
WS	1150+150	2300±300	0.0209±0.0021
WF	2210+150	310±50	0.0568+0.0045
GS	320+200	120,000±1000	0.0029±0.0001
GF	1300+150	1100±150	0.0302±0.0020

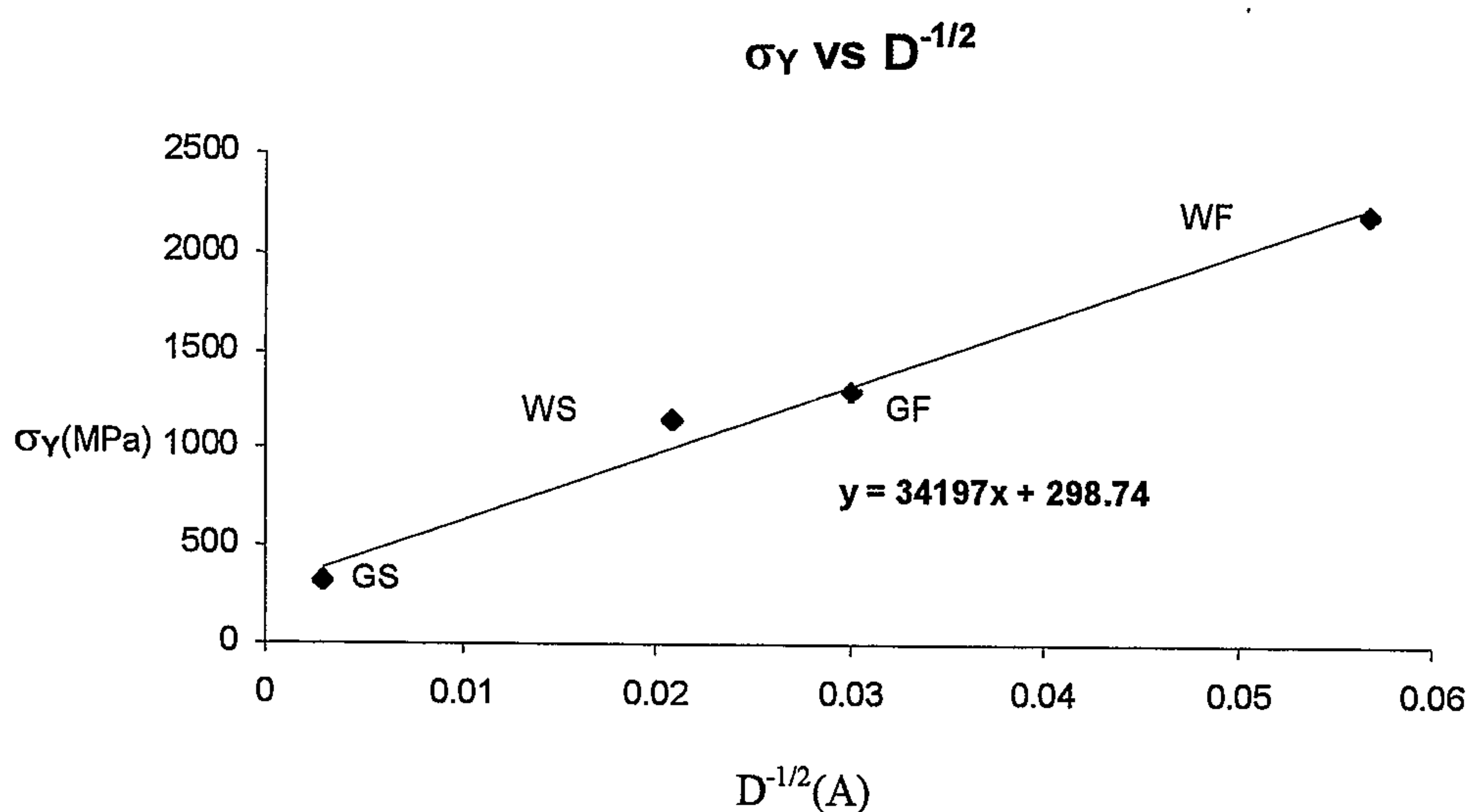


Fig.5F

A fine-grained material is harder and stronger than one which is coarse-grained. The reason is that a smaller grain size means a greater total grain boundary area to impede dislocation motion. Smaller grain size also increases the dislocation density to work-harden the structure. [5.30, 5.31, 5.32]

The TEM results also indicate that the shape of most grains in WS and WF wires are elongated. The grains from the core of the wires are less elongated in comparison to those on the shell of the wires. The reason for the different shapes of the grains are due to the higher localised shearing forces at the die/wire interface. Thus, during the wire drawing process, the subgrains which are closer to the die wall have more elongated subgrains shape. On the other hand, the subgrains in the core of the wires have less elongated subgrains as they receive less shearing force. For the GF wires, the reason for the subgrains shape looking similar in the shell and in the core of the wires is probably due to the annealing recovery the elongated grains.

The elongated grains will introduce the anisotropic properties of the metal such as the fact that the strength and plasticity along the elongated grains are higher.[5.32]

The grain shape of GS wires have a hexagonal shape in the core and the shell of the cross section of the wires and in the longitudinal section of the wires. This is probably due to the high temperature annealing used to return the deformed elongated subgrains back to their original grain shape.

5.4.2. Twinning:

The TEM results shows that twinning is present in the WS, GF and GS wires. Twinning is probably will also inherited during the continued deformation from WS to WF, but we cannot use TEM techniques to find twinning in WF wires since the grain size in these wires is too small (about 300Å) to allow the sample to be tilted.

The results that a high density band twin structure exists in WS, WF and GF suggest that due to the heavy deformation of the wire manufacturing process, stress is concentrated at a very high level in the wires. This concentration of stress results in the wires being unable to continue to develop drawing by slip and so a form of twinning occurs in order to continue the deformation. The shear stress for forming twinning is higher than for slip.[5.21, 5.31]

In the GS wires, the fact that we have seen a much lower density twin structure is probably due to the high temperature annealing used to reduce the stress concentration so that the wires may continue to be partly deformed by the slip mechanism. The different results for twinning between GS and

the rest of the wires actually explain the difference of ductility and strength property between these wires as found in the mechanical property tests.

The reason for GS wire being more ductile than other three wires (see tensile test results in chapter 3) is that GS wires possess less density of twinning and hence more slip systems than do the other three wires. Also, more slip systems existing in the GS wire implies less stress concentration and hence lower strength for the GS wires.

In addition, we should note that twinning may introduce micro-cracks at the intersection of the twins.[5.34] Thus, twinning can sometimes cause materials to be easy to fracture. This is one of the reason why WF, WS and GF wires are more easy to break than the GS wires.

5.4.3. Small angle boundaries

The diffraction spots elongated in WS,WF and GF wires results show that small angle boundaries exist in the WS, WF and GF wires. As we mentioned in chapter 2, small angle boundaries actually significantly strengthen the wires[5.30, 5.35, 5.36].This is another reason why yield strength in these three wires are much higher than the GS wires.

Small-angle boundaries can be produced in a number of ways. [5.37] They may be produced during crystal growth, during high-temperature creep deformation, or as the result of a phase transformation. The TEM results are consistent with the XRD asymmetrical experimental results (chapter 4)

which show that there is always some angle between the 111 austenite and 110 martensite grains.

5.4.4 Martensitic transformation:

As we mentioned in the introduction to this chapter and in chapter 2 (see 2.8.2.1), with this type of material and manufacturing process, the ϵ -martensite and α -martensite can be formed during the deformation process. In fact, using TEM and XRD techniques, we only found α -martensite (see 4.2.1 & 5.3.2). This can be explained as follows:

ϵ -martensite is an intermediate product between austenite and α -martensite phase transformations. When the volume of ϵ -martensite rises to 5%, this ϵ -martensite will transform to α -martensite[5.2]. The fact that we have not found ϵ -martensite in any of the orthodontic wires we researched is probably due to the heavy deformation having caused a transformation of ϵ -martensite to α -martensite.

Ferrite (δ -Fe) existed in the wires as a residual product of the high temperature heat treatment. Since its lattice parameter values are approximately those of α -martensite, it is almost impossible to distinguish these two kinds of phases using diffraction patterns.

For the chromium carbide (Cr_{23}C_6 , Cr_7C_3) and iron carbide (Fe_3C) phases, because the amount of these phases is very small, XRD techniques could not detect them. Also, because of the serious deformation structure, the grain boundaries become obscure and so since these carbides normally exist along

the grain boundaries and moreover in very small amounts, it is also very difficult to find them using TEM techniques.

As we described in chapter 2, the transformation of austenite to martensite by a diffusionless shear-type transformation in steel is one of the most common strengthening processes used for engineering materials. The reason for martensite strength of materials is that martensite has many strong barriers to dislocation motion. These barriers are provided by the fine twin structure or the high dislocation density. [5.30]

5.5 Conclusions

Four 302 type orthodontic wires produced in Australia and in Germany having the same composition (see chapter 2 for details). Since the four wires have different manufacturing processes, there are some differences in their microstructures. From the TEM investigation, it can be concluded

- The sub-grains and grain's sizes from highest to lowest for the four wires are:

$$WF < GF < WS < GS.$$

- The sub-grain shape in WF, GF, and WS are all elongated. The grain shape in GS wires are hexagonal. This could due to the high temperature annealing for the GS wire during the manufacture process.
- Twinning structures were found in all four wires (WF, WS, GF and GS). The density of twinning in GS wires was, however, much lower than in the other three wires.

- Small angle twist boundaries exist in WF, WS, and GF wires, but could not be found in GS wires.
- (111) austenite and (110) martensite texture exist in all four wires (WF, WS, GF and GS).
- Both austenite and martensite phases exist in the four wires (WF, WS, GF and GS).

5.6 Further Research proposals

The following TEM image and SAD pattern are taken from the WF wire and WS wires, respectively. These TEM pictures can provide the very interesting microstructure information to further understand the wire properties. The further TEM research recommendations are as follows:

1. Fig 5.59 is the bright field image taken from the cross-section WF wires. The parallel lines in one of the grains are probably due to the twinning or stacking faults. To identify which of the two it is, examination of diffraction patterns for this grain is needed.
2. Fig. 5.60 is the SAED pattern taken from the cross section WS wires. The elongated spot could be due to the small angle twist boundary as these deflection spots array like the circles around the centre spot. The lines between the spots may result from imperfect ordering, stacking faults, micro-twins, or layer structure. To identify which one it is, high resolution imaging and analysis is required.
3. Fig. 5.61 is the SAED pattern taken from the cross section of WS wires. The line between the spots could be due to the imperfect ordering,

stacking faults, micro-twins, or layer structure. To identify which one it is, we should undertake high resolution analysis.

4. Fig. 5.62 is the SAED pattern taken from the WS wire cross-sections. The weak spots adjacent to the bright spots could be due to the ordering, twisting, double diffraction, or two different phases. To identify what is the cause, we need to take the dark field image for those weak spots which are adjacent to the bright spots.

5. Fig. 5.63 is the SAED patterns taken from the WS wire cross-sections. The weaker spots between bright spots could be due to the ordering, twisting, double diffraction, or two different phases. To identify what is the cause, we need to take the dark field image for those weak spots which are between the bright spots.

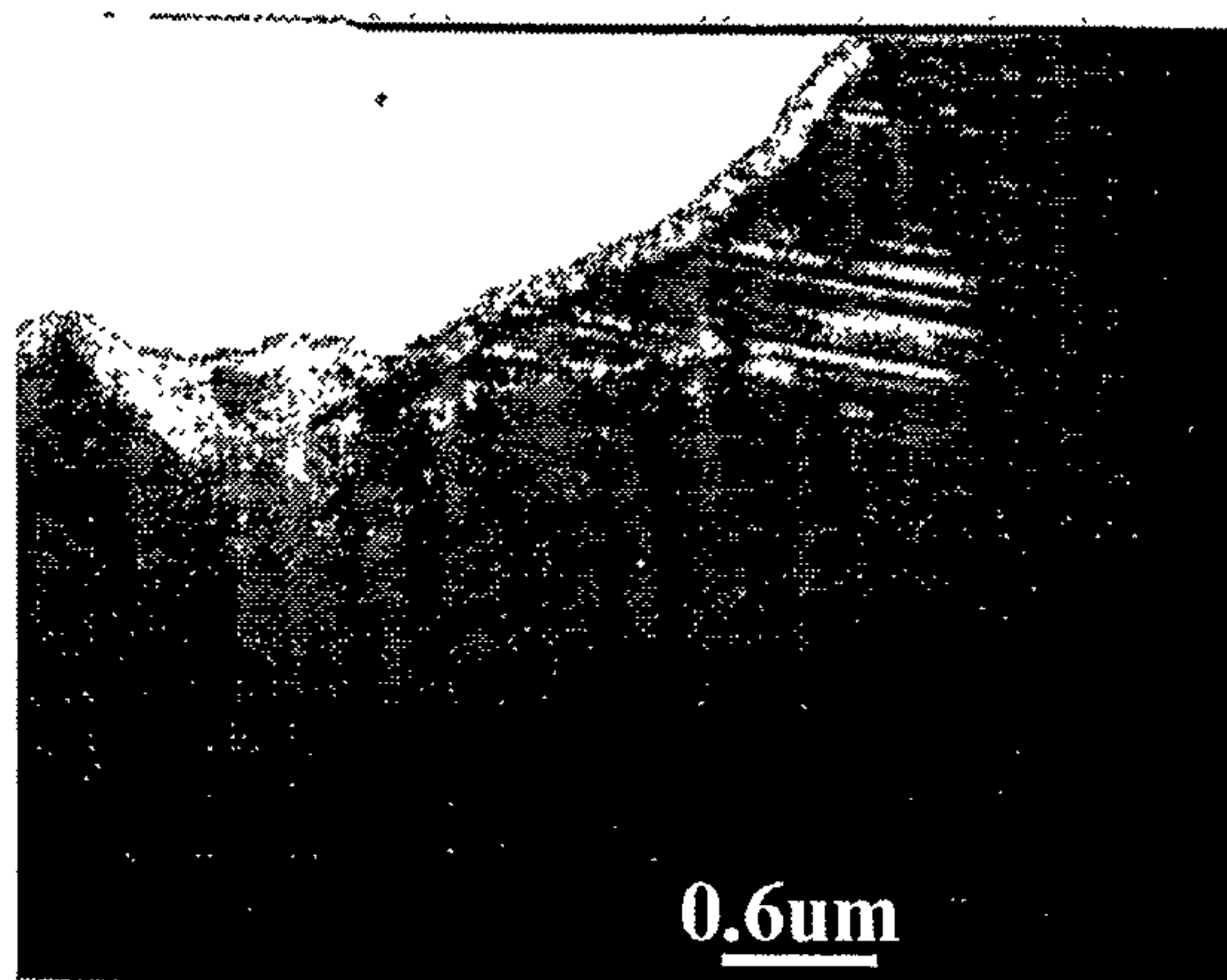


Fig. 5.59

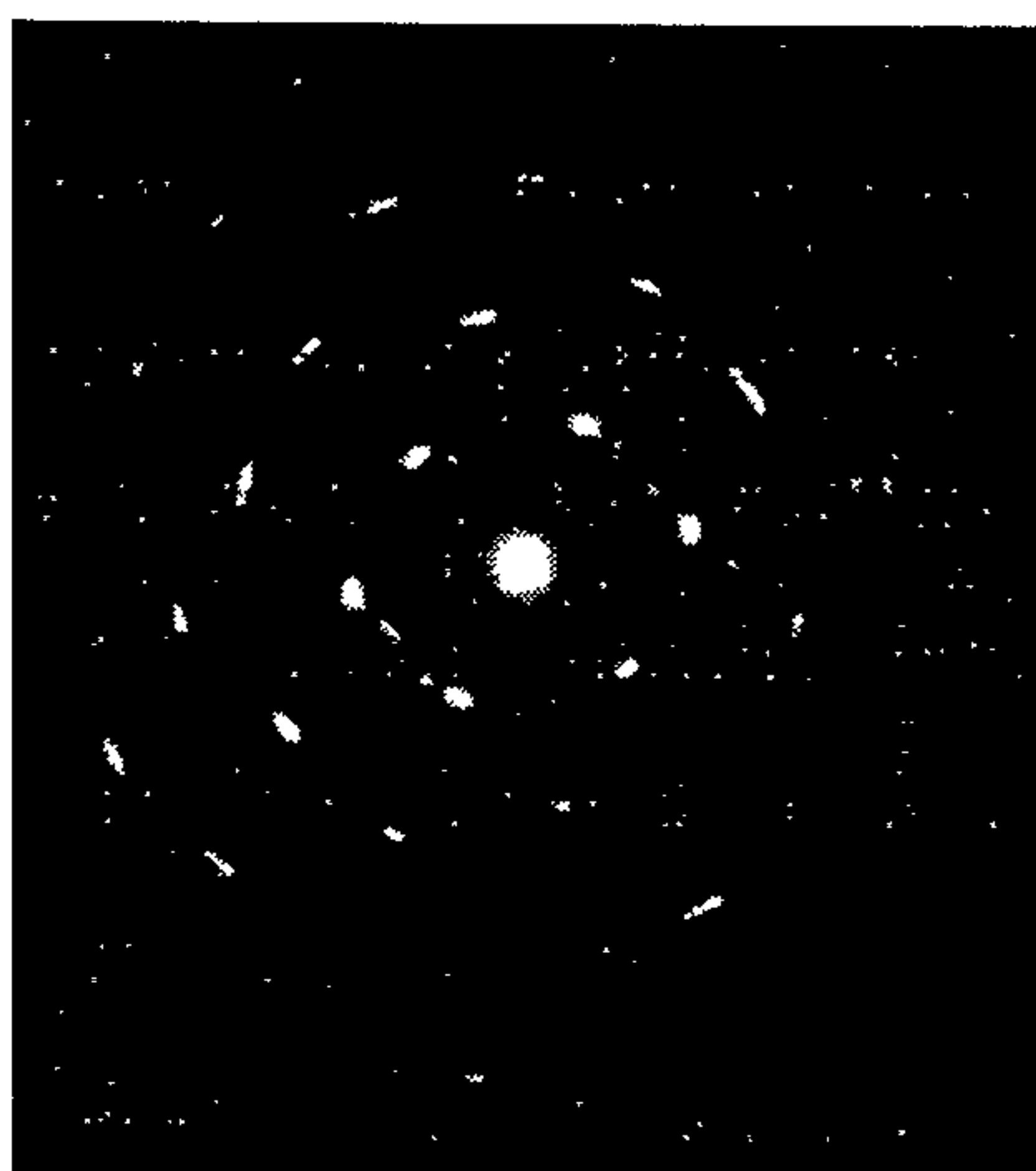


fig. 5.60

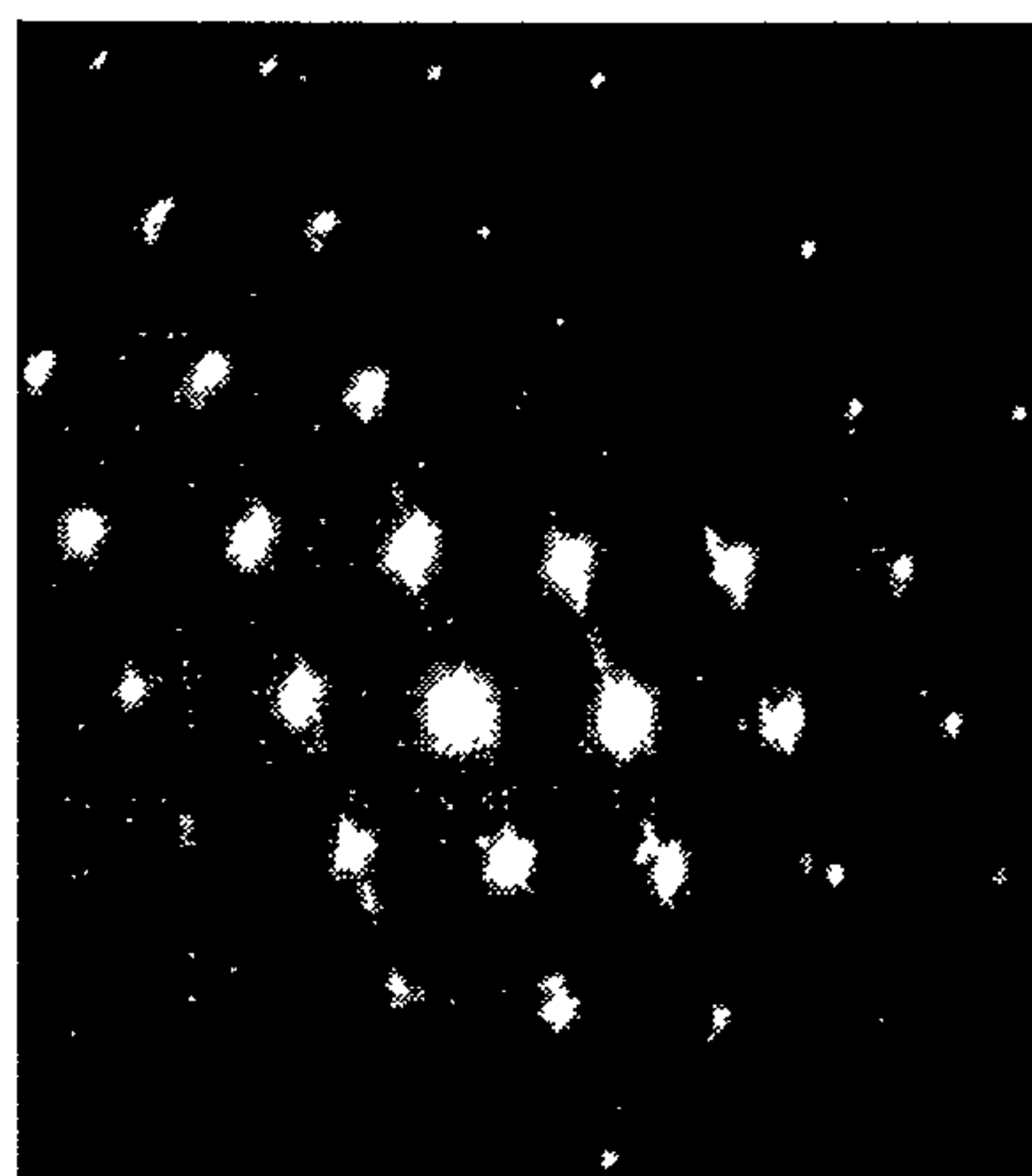


fig. 5.61

Fig.5.59 BF image taken from the cross section WF wires.

Fig.5.60 SAED pattern taken from the cross section WS wire.

Fig.5.61 SAED pattern taken from the cross section WS wire.

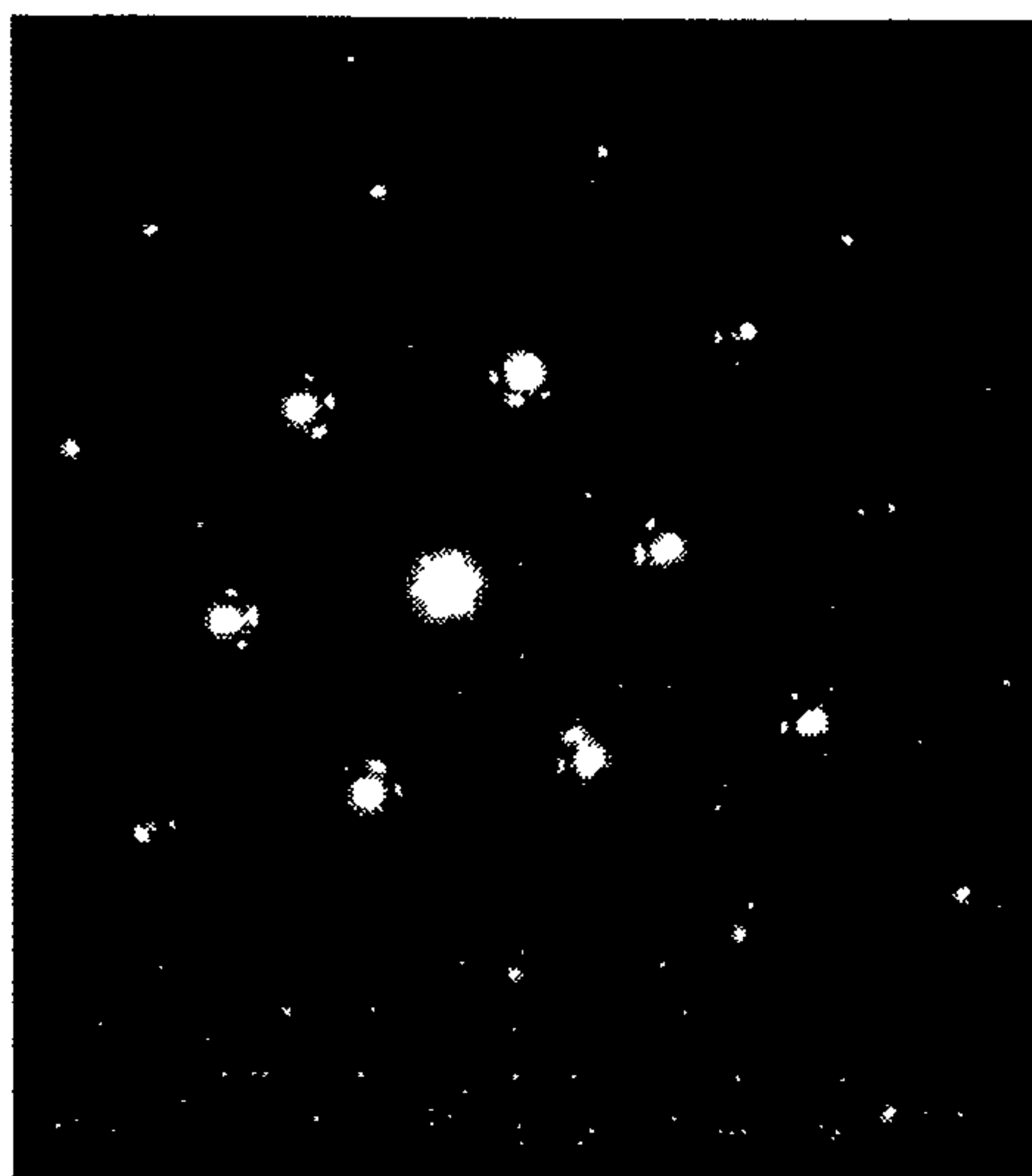


Fig. 5.62

Fig.5.62 SAED pattern taken from the cross section WS wire.

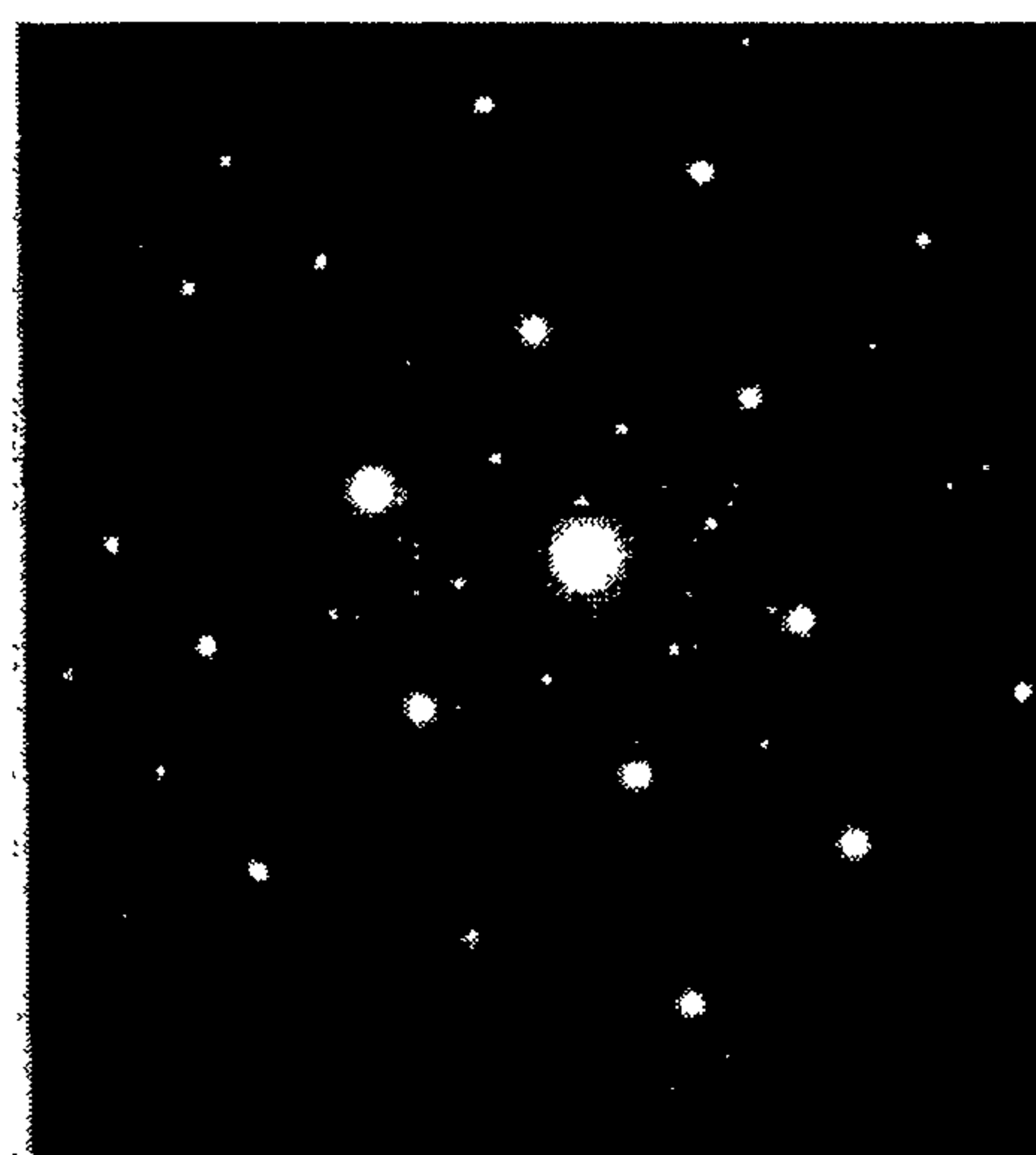


fig. 5.63

Fig.5.63 SAED pattern taken from the cross section WS wire.

Appendix 5.1: The SAED diffraction zones and respective scale of X,Y axis of the double tilt holder for Fig.5.27, 5.28,and 5.29:

Zones	X axis(degree)	Y axis(degree)
[110]	-10	-2
[211]	2	29
[010]	28	-28.5

Appendix 5.2. The respective experimental and theoretical inter-zones between the zones for Fig 5.27, 5.28, and 5.29.

Zones	Experimental inter-zone values	Theoretical inter-zone values
$[110]^\wedge [211]$	$33^\circ \pm 5$	30°
$[211]^\wedge [010]$	$61^\circ \pm 5$	$65^\circ 54'$
$[110]^\wedge [010]$	$45^\circ \pm 5$	45°

References of chapter 5

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Chapter 6 Summary and Conclusions.

In the previous chapters, the main examination has been of the microstructure and mechanical properties of stainless steel wires using TEM, XRD, SEM and mechanical property test techniques. In this chapter we summarise and combine the conclusions that can be drawn from the above experiments and their applications.

Firstly, the conclusions from the mechanical properties analysis are:

- The elasticity modulus measured in the tensile tests using an extensometer on the four as-received wires and the set of annealed WF wires were all approximately 200GPa. This result is contrary to the results by other researchers but consistent with the principle from materials theory that the elastic modulus, E , depends only upon the type of the materials and that wire drawing processes, such as cold-working or heat-treatment, should not reduce the value of E .
- The elasticity modulus measured in the tensile tests without using an extensometer was about 170GPa, which is 15% lower than the value from tensile tests using the extensometer (which is also the standard value). The difference between the results with and without an extensometer for the same wires suggests that the lower E value observations were due to the machine compliance error.
- The E value measured in 3-point bending and 4-point bending tests were all about 170GPa, which is also 15% lower than the value in the standard value. The reason for this lower E value in flexure tests may due to the fact that an extensometer cannot be used.

- The tension test results suggest that the 0.1%YS are different in the four experimental wires although they are of the same composition. The order from higher to lower for 0.1% YS value is:

$$WF > GF > WS > GS$$

- For the same wire, different 0.1% yield strength values were found using 3-point bending tests, 4-point bending tests and tensile tests. These differences can be explained by the different nature of the deformation modes.
- The tensile tests and the SEM micrographs of tension and flexure tests suggested that the GS wires are considerably more ductile than the other three wires and that the WF wires are the most brittle.

The conclusions for microstructure analysis are:

- The subgrain and grain's sizes from highest to lowest for the four wires are:

$$WF < GF < WS < GS.$$

- The subgrains shape in WF, GF, and WS are all elongated. The grain shape in GS wires are hexagonal. This could due to the high temperature annealing for the GS wire during the manufacture process.
- Twinning structures were found in all four wires (WF, WS, GF and GS). The density of twinning in GS wires was, however, much lower than in the other three wires.

- Small angle twist boundaries exist in WF, WS, and GF wires, but could not be found in GS wires.
- (111) austenite and (110) martensite texture exist in all four wires (WF, WS, GF and GS). The measure of these textures for the four wires was obtained from XRD analysis and presented in table 4.4.
- Both austenite and martensite phases exist in the four wires (WF, WS, GF and GS). The measure of these phases for the wires were obtained through XRD analysis and presented in table 4.4.

Combining the above mechanical properties and microstructure results, the following conclusions can be drawn based upon standard results from material science and metallurgy theory as presented in Chapter 2.

Since 0.1% YS and E are the two mechanical property parameters which are the most closely related to the clinical effectiveness of the wires and since all the wires researched in this study are 302 stainless steel, the E value for all these wires are same. Therefore, the microstructure study gives more insight into differences in strength. Brittleness is another important mechanical property parameter and the difference of brittleness between these wires can also be explained by the microstructure:

- Grain size ordering is $GS > WS > GF > WF$, this is precisely the opposite to the tensile 0.1% yield strengths ordering which is $GS < WS < GF < WF$. The relationship between strength and grain size fits well with the Hall-Petch equation.

The reason is that a small grain size means a greater total grain boundary area to impede dislocation motion. Therefore, a fine-grained material is

harder and stronger than one which is coarse-grained. Smaller grain size also increases the dislocation density, thereby work-hardening the structure.

- The subgrains in WF, WS and GF are elongated. This introduces anisotropy properties in these wires as more grain boundaries lie in one direction.
- Twinning densities in WF, WS and GF wires were much higher than in the GS wires. This microstructure result corresponds with the results of the mechanical property tests which show GS wire to have a much lower strength than the other three wires.

The reason for this is that during the heavy deformation wire drawing process, the concentration of stress results in the wires being unable to continue to be drawn by slip and so a form of twinning occurs in order to continue the deformation. The shear stress for twinning is higher than for slip.

It is anticipated that twinning also causes the wires to break in the clinical application. The reason is that as a twin grows in size, the accommodation normally requires plastic deformation in the surrounding metal. If the matrix is not able to accommodate the twinning shear, then a hole may develop at the point where the accommodation was not obtained. This hole can become the initial source of a crack. This may be the reason why the wires with less twinning density (i.e. GS wires) are less easy to break than the other three wires.

- Small angle boundaries exist in WF, WS and GF wires but not in GS wires. This microstructure result also explains the reason why WF, WS and GF wires have much higher strength than GS wires since the small angle boundaries significantly increase the strength of the wires.
- (111) austenite texture and (110) martensite texture exist in four wires. This kind of texture suggested that more slip planes form perpendicular to the wire axis (the cross section of the wires). These slip planes are (111) in fcc austenite structures and (110) in bcc martensite structures. However, fewer slip planes form parallel to the wires axis (the longitudinal section of the wires).

Since the cubic metal can only deform on slip planes and along the slip directions, this texture makes the wire hard to deform in the longitudinal direction (the wires axis). Therefore, this kind of texture actually causes the deformation process to be more difficult along the wire axis, so, the strength of the wires is increased.

- From the XRD quantitative analysis, α -martensite phases exist in the four wires, the amounts are:

GF: 82%; WF: 74%; GS: 70%; WS: 56%.

Martensite transformation in steel is one of the most common strengthening processes used for engineering materials. The reason for martensite strengthening of materials is that martensite has many strong barriers to dislocation motion. These barriers are provided by a fine twin structure or a high dislocation density.

Martensite phases also can result in some brittleness for the wires.

However, the yield strength and brittleness of these four wires is not exactly proportional to the amount of martensite phase. This is because yield strength is affected by the combination of the factors mentioned above and not only the amount of martensitic phase.

The list of the publications and the conference papers.

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