

## 10. SCANNING ELECTRON MICROSCOPY

The surface of a specimen has been defined by Plueddemann (1982) as, "the region from which information is received in a particular measurement". Under this definition, surfaces may be examined by exposing the surface to a variety of controlled radiations and examining the emitted radiation by instrumental techniques. Optical microscopy is of limited value in examining the interface region, since resolution is limited by the wavelength of visible light to about 10,000 Å. By using an electron beam for imaging, it is possible to obtain resolution down to about 100 Å. Scanning electron microscopy is therefore used to study details of fracture surfaces, but cannot resolve to monomolecular coverage (Plueddemann 1982 p.76).

High-energy electron beams from the scanning electron microscope penetrate the solid and cause emission of X-rays and Auger electrons. An Auger electron is an electron characteristic of the de-excitation energy of the surface. Auger electrons may be controlled to escape depths as shallow as 3 to 6 Å in some materials and are more useful than X-rays for surface analysis (Plueddemann 1982 p.76).

Unfortunately, although surface and interface morphology can be determined with scanning electron microscopy, no deductions can be made concerning bonding (Beech 1982). Other methods which may be of assistance include Laser-Raman spectroscopy, X-ray photoelectron spectroscopy and secondary-ion mass spectroscopy (Kinloch 1980).

## **11. MATERIALS AND METHODS**

### **11.1. INTRODUCTION**

The bonding agents selected for this study were chosen as a result of the manufacturers' claim that they could bond to dental gold alloy. These materials were Fusion (George Taub Products & Fusion Co. Inc.), Scotchprime (Minnesota Mining and Manufacturing Co.), and Panavia Ex (Kuraray Co.). Silar (Minnesota Mining and Manufacturing Co.) restorative material was the composite resin used with both Fusion and Scotchprime due to the insistence by the manufacturers of Scotchprime that 3M products were only compatible with each other. A chemical-curing composite was essential as the stainless steel lingual buttons would not permit the passage of light for light-curing adhesives.

An aim of this experiment is to determine whether a clinically improved bond strength can be obtained by the use of a commercially available gold bonding agent or whether, as is already practised, (Norton 1987) simply roughening the gold surface in combination with an enamel-bonding material produces sufficient bond strength and is therefore more cost-effective.

### **11.2. BONDING MATERIALS**

#### **11.2.1. FUSION** (see Figure 7)

According to the manufacturer's promotional literature Fusion chemically bonds any combination of metal to metal, as well as gold to composite. It is supplied as an aqueous silane primer under acidic conditions, capable of use with microfilled composites such as Silar. Unlike Scotchprime, it is sold in the unhydrolyzed state as a two part system and therefore the



Figure 7 Fusion bonding kit.



Figure 8 Scotchprime bonding kit.



Figure 9 Panavia Ex adhesive system.

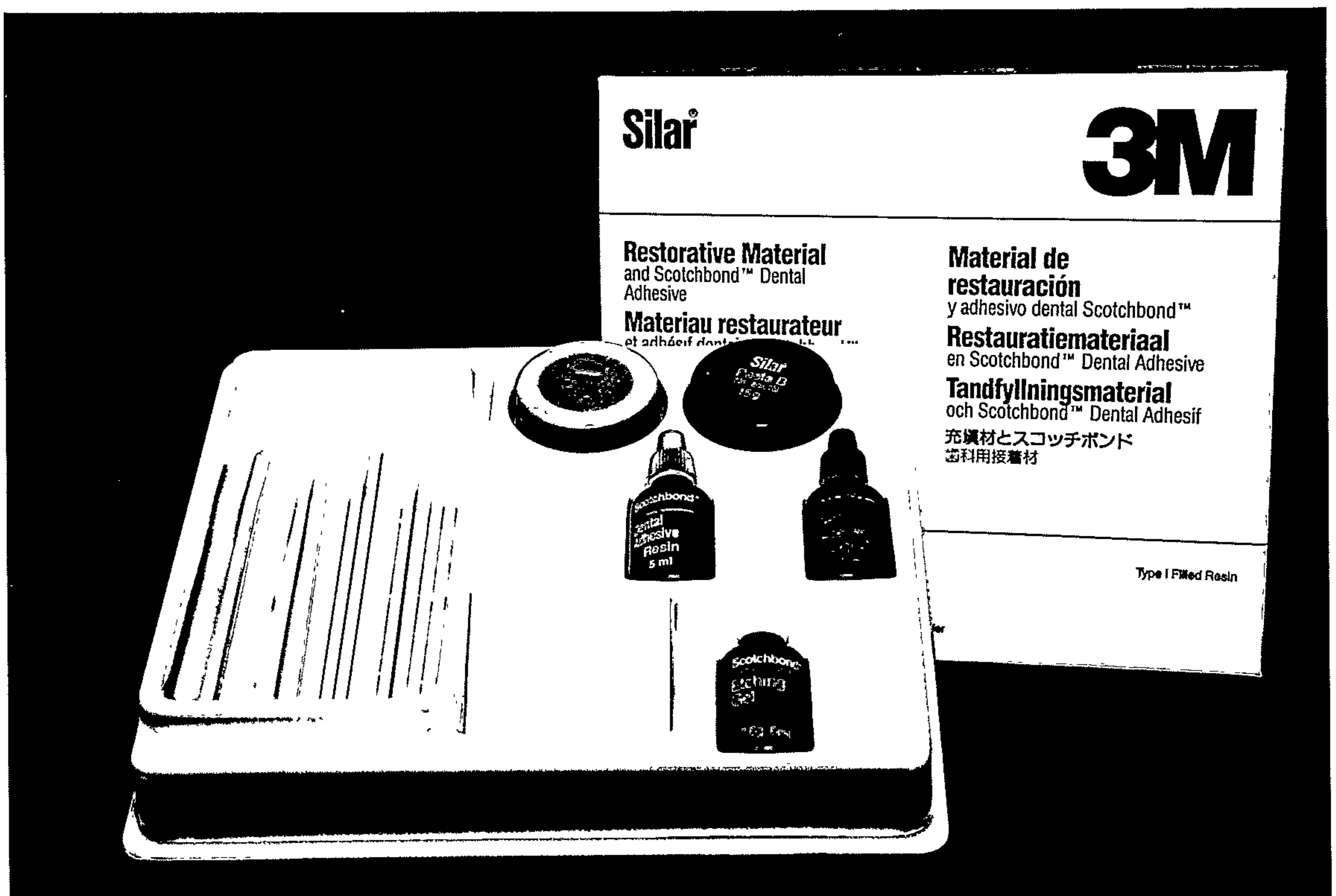


Figure 10 Silar composite restorative kit including Scotchbond chemical curing adhesive.

operator is required to activate the solution fifteen minutes prior to use. Its shelf life is claimed to be one year.

The nature of the silane bond that Fusion creates is one to the oxides of the substrate (Taub 1987). To supplement this the manufacturer recommends that the metal surface be roughened prior to application of Fusion, to enhance the bond by increasing the surface for bonding, as well as providing some mechanical retention. The manufacturer also stresses the importance of acid-etching the metal before applying the Fusion as this increases the flowability of the silane.

The manufacturer points out that Fusion's bond to non-precious metal is greater than to precious metal. This bond strength also requires 24 hours to fully mature.

Fusion has been used in previous studies by Eames, Rogers, Feller and Price (1977), Ghassemi-Tary (1979), Highton, Caputo and Matyas (1979), and Azarbal, Boyer and Chan (1986).

#### **11.2.2. SCOTCHPRIME** (see Figure 8)

Scotchprime is supplied as a dilute solution of 100% activated  $\gamma$ -methacryloxypropyltrimethoxysilane in ethyl alcohol.

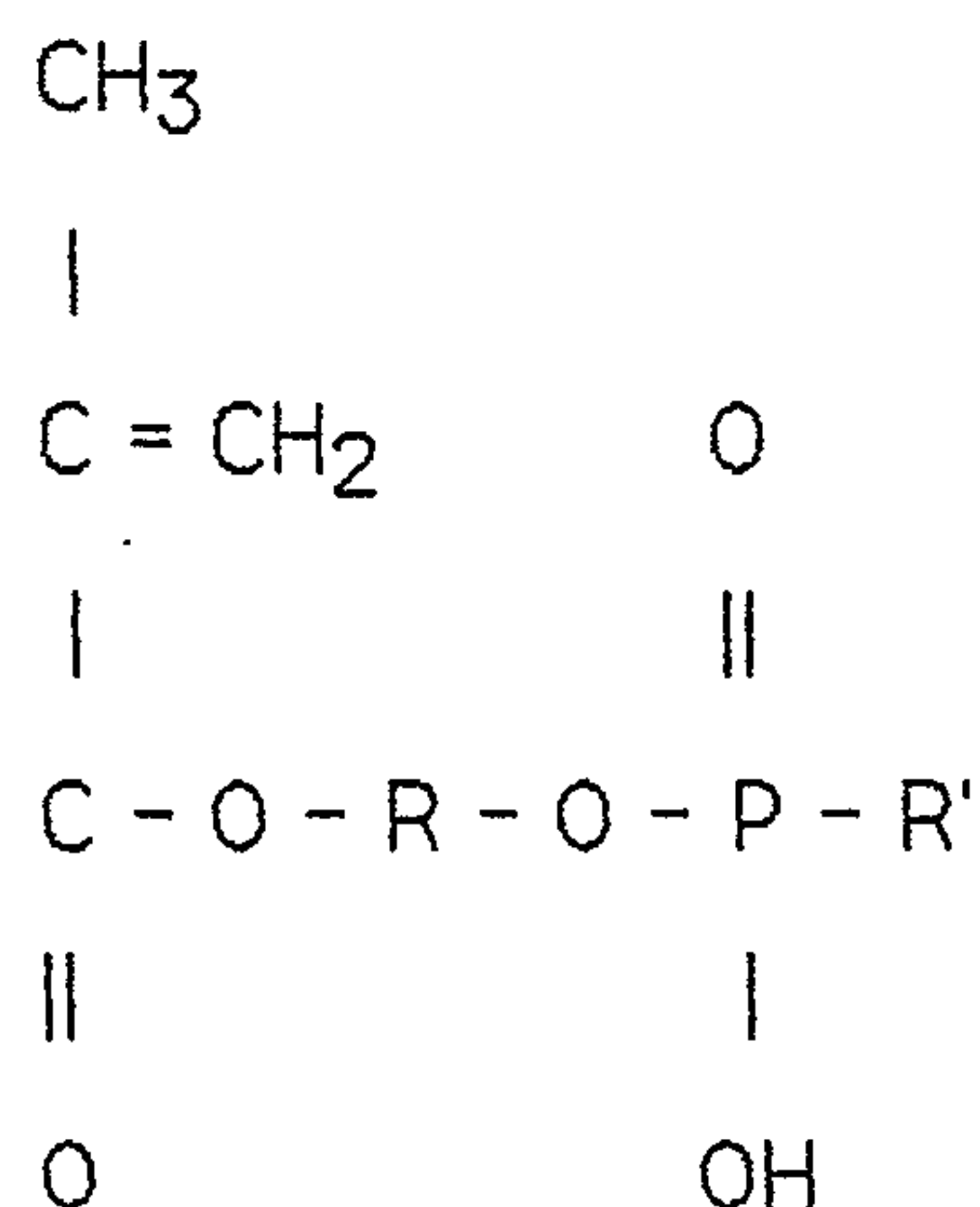
The manufacturers' claim that in using this product to bond to porcelain, the bond strength exceeds the strength of the porcelain. However, they cannot make the same claim for gold, citing the bond to gold alloy as being equivalent to the bond of Scotchbond adhesive to dentine. According to the promotional literature available, Scotchprime chemically unites with the metal oxides on the alloy surface to give improved, water-resistant bonding. The manufacturer also advises that the gold surface be roughened prior to bonding to take advantage of any mechanical retention.

Scotchprime has been used in the study by Stokes, Hood and Tidmarsh (1986).

### 11.2.3. PANAVIA EX (see Figure 9)

According to the promotional literature, Panavia Ex is a type of composite resin supplied as a liquid and a powder. The powder consists of fine particle inorganic fillers which after mixing constitute 76% of the material. The liquid is a bi-functional metha-acrylate monomer mixed with adhesive monomer containing a phosphate ester group (Omura, Yamauchi, Harada & Wada 1984).

The formula for the adhesive monomer of Panavia Ex is;



Setting of Panavia is initiated by the formation of radicals which are generated when the reduction agent contained is mixed with peroxide. The radicals so formed bind densely with the dimetha-acrylate monomer to commence curing. Therefore, cured Panavia consists of a three dimensional compound of resin matrix containing fine glass filler particles.

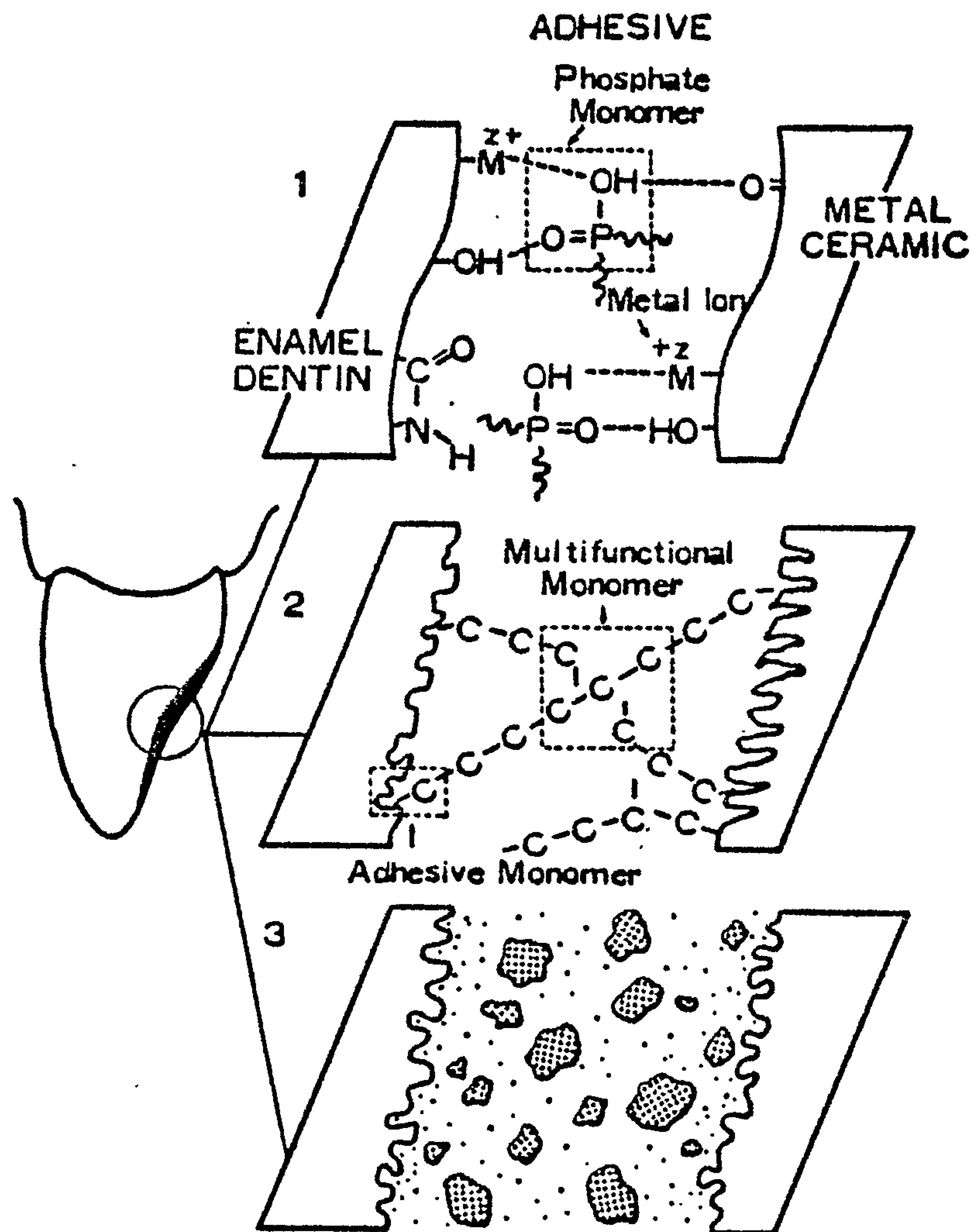


**Figure 11** Bonding mechanism of Panavia Ex monomer with metal-ceramic and human dentine (Promotional literature).

The manufacturer claims that Panavia Ex bonds to dental gold alloys. This bond is greater to a metal with an oxide film. The resin bonds to the metal oxide via the phosphate ester group (see Figure 11). The manufacturer claims that Panavia's bond to sandblasted gold alloy is nearly 40% greater than its bond to etched human enamel. The manufacturer advises etching of the gold prior to metal bonding.

Omura, Yamauchi, Harada & Wada (1984) described the cement as having twice the compressive strength of zinc phosphate and polycarboxylate cement. Panavia Ex has been used in studies by Thompson, Grolman and Liao (1985).

Panavia Ex was selected for shear bond testing as it is currently in use clinically as a metal bonding orthodontic agent (Fox 1986) but unlike Fusion and Scotchprime it is not a silane material.

#### **11.2.4. SILAR** (see Figure 10)

Strictly speaking Silar is not a composite material but rather is classified as a type-I direct filling resin (due to its higher water sorption value) in accordance with the American Dental Association's Specification No. 27.

The working time is about one and one-half minutes and the setting time of Silar is approximately two minutes from the start of the mix. The product can be finished after about 5 minutes. The shelf life of Silar at room temperature is six months. The manufacturer accepts the use of a syringe to dispense the material (Promotional literature).

The Silar paste is prepared from three main ingredients. The resin is Bowen's resin (Bis-GMA) plus diluent; the inorganic filler is finely ground silicon dioxide with an average particle

size of 0.04 microns; and the organic filler is finely ground particles of polymerized composite. The organic filler constitutes 52% by weight of the Silar. The silicon dioxide is silane-treated to improve the bond between the filler and the resin and its small size confers a smoother finish compared to composites with larger particle size. The manufacturer stresses the need to acid-etch the bonding surface with 3M's 37% orthophosphoric acid and apply a sealant, prior to use of Silar, to prevent microleakage.

Stark (1985) notes that finishing of Silar should be delayed for five minutes after setting.

Silar is accepted as a resin-based restorative material under Australian Dental Standard 1278-1982, as recorded in the 1984 list of certified products.

#### **11.2.5. SCOTCHBOND** (see Figure 10)

This is the bonding agent packaged with Silar restorative material. 3M warns that the retentive strength of Scotchbond used with composite resins manufactured by other companies will not be as great as when used with 3M composite resins (Stark 1985). However, Chan, Reinhardt and Jensen (1984) and Chan, Reinhardt, and Boyer (1985) found that the retentive strength of Scotchbond did not differ significantly when used with other resins.

#### **11.3. DEGUDENT U**

Degudent U is a pale yellow, extra hard ceramic alloy. It is composed of 96.1% gold and platinum metals, of which 77.3% is gold. It contains 1.2% silver and 2.7% base metals. It has a

melting range of 1150-1260°C and a casting preheating temperature requirement of 850°C.

Nally (1968) noted that this was the most complex of the dental gold alloys in his study. Degussa Australia Limited, the manufacturer's of Degudent, recommend this alloy for use in bridgework of any physiologically acceptable span length. It is the alloy of choice for all crown and bridgework constructed in The United Dental Hospital of Sydney (Powder 1987).

Degudent U was the gold alloy used by Stokes, Hood and Tidmarsh (1986) in their study on orthodontic bonding to gold.

#### **11.4. SPECIMEN STORAGE**

To correlate adhesion test data with expected clinical behaviour, the test specimens should be stored in an environment simulating the oral cavity (Lee, Swartz & Culp 1969). Beech (1978) states that to determine those adhesives with clinical potential, bonds should be tested after a period of water immersion.

To simplify matters, specimens are generally stored in water (Low, Davies & von Fraunhofer 1975b; Omura, Yamauchi, Harada & Wada 1984; Knoll, Gwinnett & Wolff 1986) or artificial saliva (Johnson, Hembree & Weber 1976; Moser, Marshall & Green 1979) for a specified period of time before measurements are made. Other storage media include deionized water, distilled water, and 10% formalin (Alexandre, Young, Sandrik & Bowman 1981).

Distilled water at 37°C for 24 hours has been used by Eick, Johnson, Fromer, Good and Neumann (1972), Faust, Grego, Fan and Powers (1978), Dickinson and Powers (1980), Buzzitta, Hallgren

and Powers (1982), Azarbal, Boyer and Chan (1986), and Jacobs, Kuftinec, Showfety and von Fraunhofer (1986). Deionized water at 37°C has been used by Mizrahi, Cleaton-Jones & Austin (1981).

If the results of tests after simple water storage are encouraging, more elaborate setups to study adhesive strength with temperature cycling should be conducted to determine the effects of thermal stresses on the bond strength (Brauer & Huget 1972). Beech and Jalaly (1980) felt that simple immersion in water was a poor replication of in vivo conditions, but that it could serve as a useful guide, to oral conditions. However, Beech (1982) notes that even with thermal cycling, there is no guarantee of equivalent performance in the mouth.

Jassem, Retief and Jamison (1981) found that temperature cycling did not have a significant effect on shear bond strength when compared to the shear bond strength of uncycled specimens.

Accelerated aging is quite often suspect because accelerating conditions, such as temperature, may cause reactions which would not be encountered in the intended service environment (De Lollis 1980). Thompson, Grolman and Liao (1985) stated that thermal cycling had no significant effect on the bond strength of Panavia Ex cement.

Chan, Reinhardt, and Boyer (1985) found no difference in shear strengths among samples of Scotchbond dentin bonding agent which were subjected to thermal cycling, and those which were immersed in water at 37°C.

### 11.5. ORTHODONTIC BRACKETS

As adhesive systems improve and orthodontic bracket bases are reduced in size, variables such as weld spots and wire mesh size become of greater importance during bonding (Maijer & Smith 1981). (see Figures 12 & 13)

Low, Davies and von Fraunhofer (1975a), Reynolds and von Fraunhofer (1976 & 1977) and Buonocore (1981a) have shown that larger bracket mesh size generally results in higher bond strengths.

Dickinson and Powers (1980), and Maijer and Smith (1981) point out that the spot welds by which certain brackets have the mesh attached to them, not only decrease the nominal area available for bonding but also act as areas of stress concentration which can initiate cement fracture. This is of particular importance if the weld spots are located on the margins of the bracket bases where adhesive voids may occur leading to leakage and early bond loss.

Reynolds and von Fraunhofer (1976), Lopez (1980), and Dickinson and Powers (1980) showed that bond strength was independant of bracket base area.

Buonocore (1981a) notes that the bracket base should approximate the substrate. This avoids the possibility of starved joints acting as areas of stress concentration and inducing bond failure at relatively light loads.

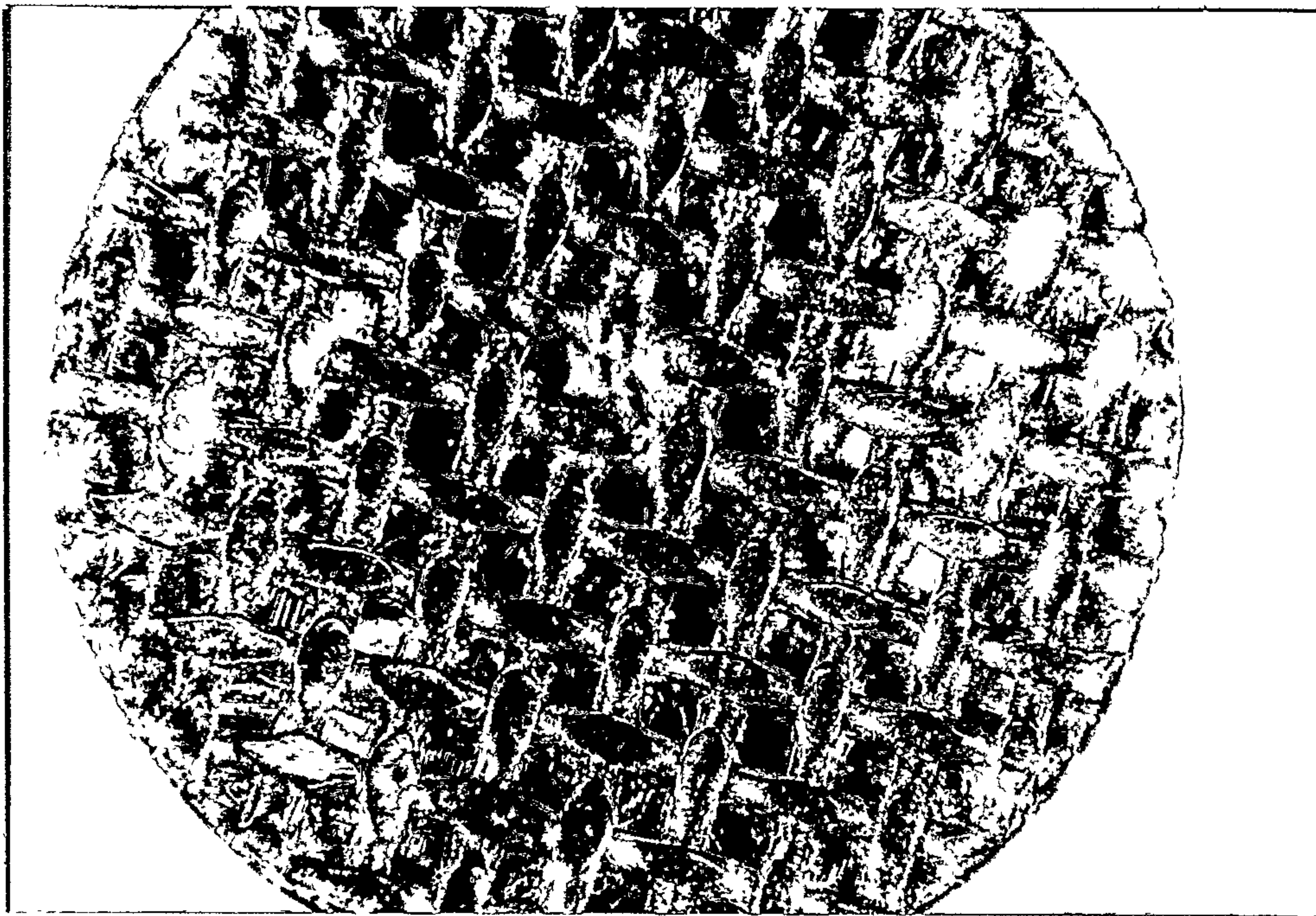


**Figure 12** Mesh surface of lingual button magnified 16 times.

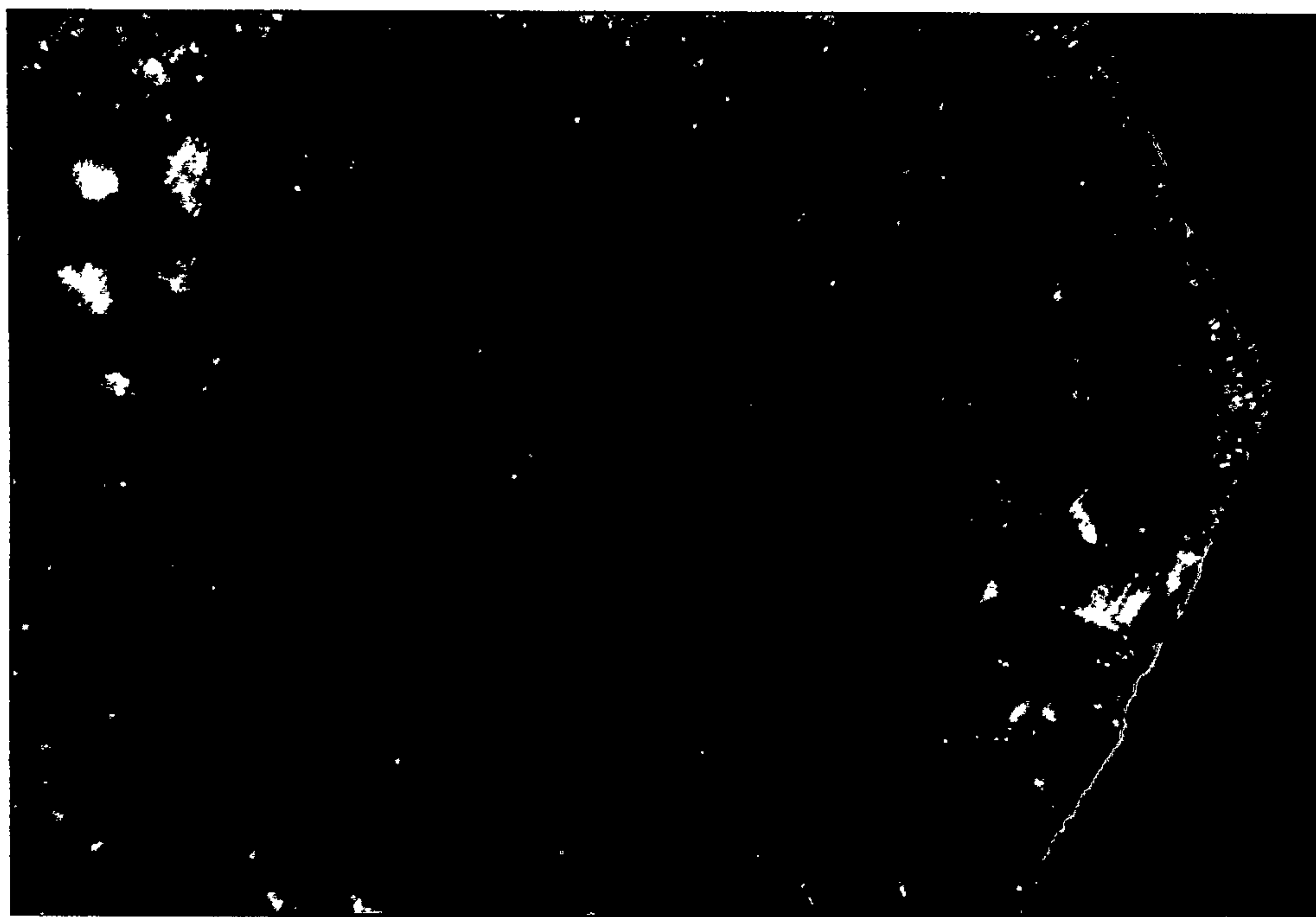


**Figure 13** Surface of lingual button following shear testing, adhesive appears to have remained attached to the mesh.

## 12. EXPERIMENT

### **12.1. CONSTRUCTION OF GOLD SURFACE**

80 gold alloy squares were constructed, six millemetres square by a half millemetre thick, from denture relief wax cast in Degudent U. The wax patterns were sprued with a large reservoir to prevent porosity and invested in Ceramigold (Whip-Mix Corp.), a phosphate-bound investment (see Figure 14). The castings were placed in a furnace pre-heated to 300°C and left for 30 minutes to allow wax elimination. The castings were next transferred to another furnace for pre-heating to 850°C for another 20 minutes (see Figure 15). Sufficient Degudent U was placed in the crucible of the casting centrifuge and heated to liquidus point of 1400°C with a propane-oxygen torch and maintained at that temperature for 30 seconds prior to removal of the casting ring from the furnace and casting of gold patterns. The castings were allow to bench cool for twenty minutes before being removed from the investment.

Following removal, the castings were sandblasted to remove any remaining investment (see Figure 15) and the sprues cut with a fine Jo-dandy disc, to prevent any distortion, leaving part of the sprue attached to the pattern for retention. The gold alloy squares were then mounted in Velmix cylinders (Hirce, Sather & Chac 1980; Alexandre, Young, Sandrik & Bowman 1981; Jacobs, Kuftinec, Showfety & von Fraunhofer 1986; Knoll, Gwinnett & Wolff 1986) by positioning them centrally with a water soluble adhesive in the base of a brass mould and pouring the die-stone around them. The cylinders were allowed to set for three hours and then removed and placed in dry storage, awaiting



**Figure 14** Materials required for construction of gold alloy surfaces.

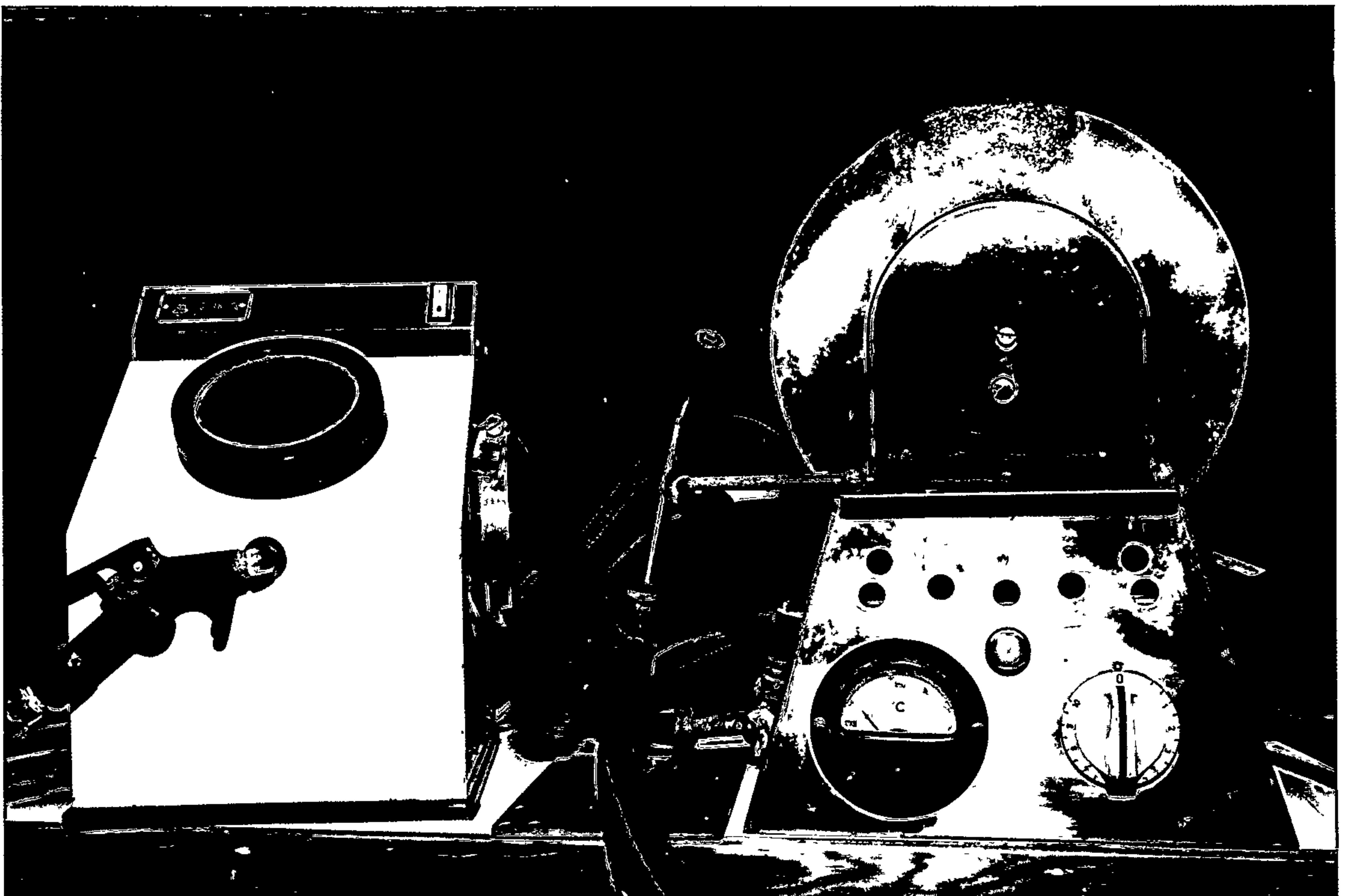


**Figure 15** Gold alloy casting furnace and sandblaster for cleaning castings.

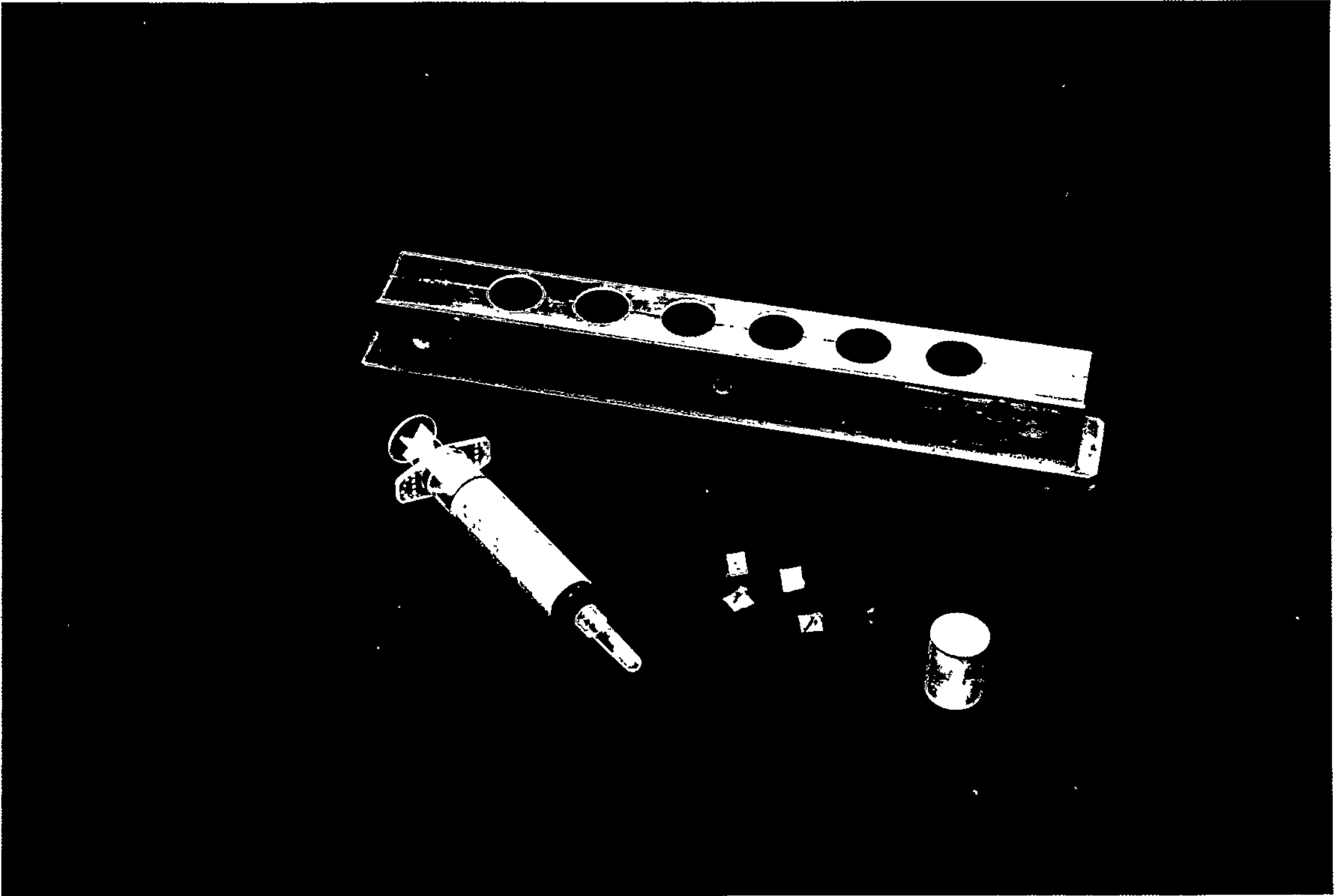


**Figure 16** Brass molding cylinder and water soluble adhesive for investing gold squares in diestone.

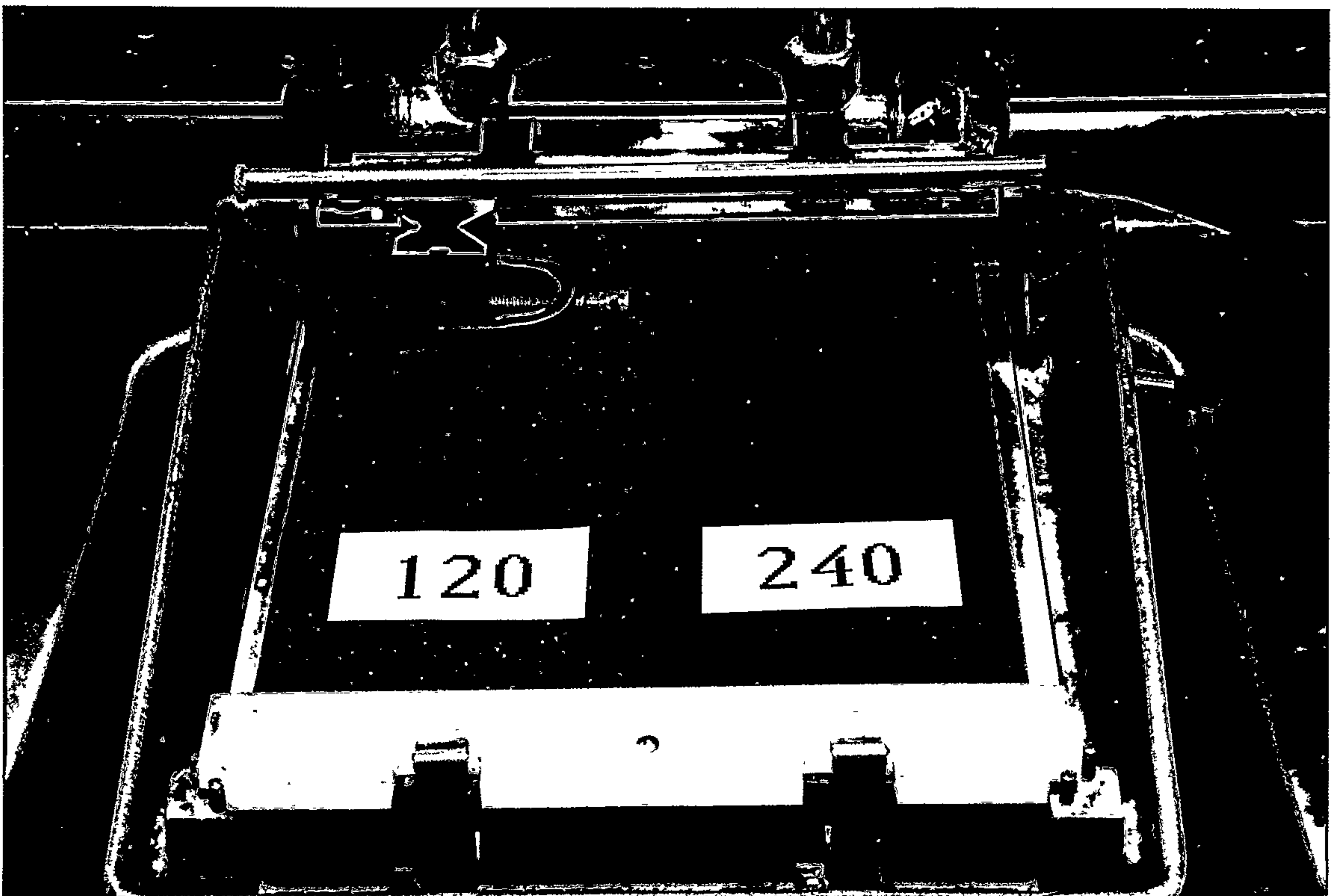


**Figure 17** Lapping tray, silicon carbide paper and V-block used to flatten and abraid gold surfaces.

further preparation (see Figure 16).

## **12.2. PREPARATION OF GOLD SURFACES**

The specimens, held in a V-block (Rogers & Griffith 1977), were sanded flat on a lapping tray (Beech & Jalaly 1980) using progressively finer grades of silicon carbide paper from 120 to 240 grit (Eames, Rogers, Feller & Price 1977). This produced a roughened (Jochen & Caputo 1977), but clinically flat (Beech 1982) surface perpendicular to the long axis of the diestone cylinder, for mounting in the shear testing machine (see Figure 17). The specimens were then examined under a Wild Photomakroskop M 400, at magnifications of 6.3 to 32 times, for surface irregularities (see Figures 18 & 19). Any necessary adjustments were made.

Next the specimens were ultra-sonically cleaned for two minutes (De Lollis 1980; Chan, Reinhardt & Schulein 1985) and the alloy surfaces washed with alcohol prior to bonding procedures. All surfaces were then isolated with a self-adhesive masking ring (contact adhesive) with a punched-out central area of approximately 3.5 mm. diameter to ensure a uniform area for bonding (Low, Davies and von Fraunhofer 1975b; Beech & Jalaly 1981; Bassier 1984). Any punched-out areas which did not appear centrally located were rejected (see Figure 20).

## **12.3. BONDING PROCEDURE**

The cylinders were numerically engraved and lingual buttons (TP Laboratories Inc. : Order No. 224-112, Batch No. 0557000) of approximately 3.5 mm. diameter bonded in the following groups:

|             |              |            |
|-------------|--------------|------------|
| Scotchbond  | Nos. 31 - 40 | 10 minutes |
|             | Nos. 61 - 70 | 24 hours   |
| Scotchprime | Nos. 11 - 20 | 10 minutes |
|             | Nos. 1 - 10  | 24 hours   |
| Fusion      | Nos. 71 - 80 | 10 minutes |
|             | Nos. 21 - 30 | 24 hours   |
| Panavia Ex  | Nos. 51 - 60 | 10 minutes |
|             | Nos. 41 - 50 | 24 hours   |

The times listed refer to the period elapsed from initial button placement to de-bonding (see Figure 20). Lingual buttons were used because of reports of consistent bond strength and reduced possibility of deformation during testing (Jacobs, Kuflinec, Showfety & von Fraunhofer 1986). The buttons were all visually examined prior to bonding and any with welding imperfections were discarded.

Each of the materials was used in accordance with the manufacturer's instructions (see Figure 21).

In the case of Scotchbond this meant:-

1) Prophylaxis of the exposed area of the alloy, using a flour of pumice and water, for 5 seconds with a rubber cup in a low speed handpiece (Gorelick, Masunga, Thomas & Zachrisson 1978).

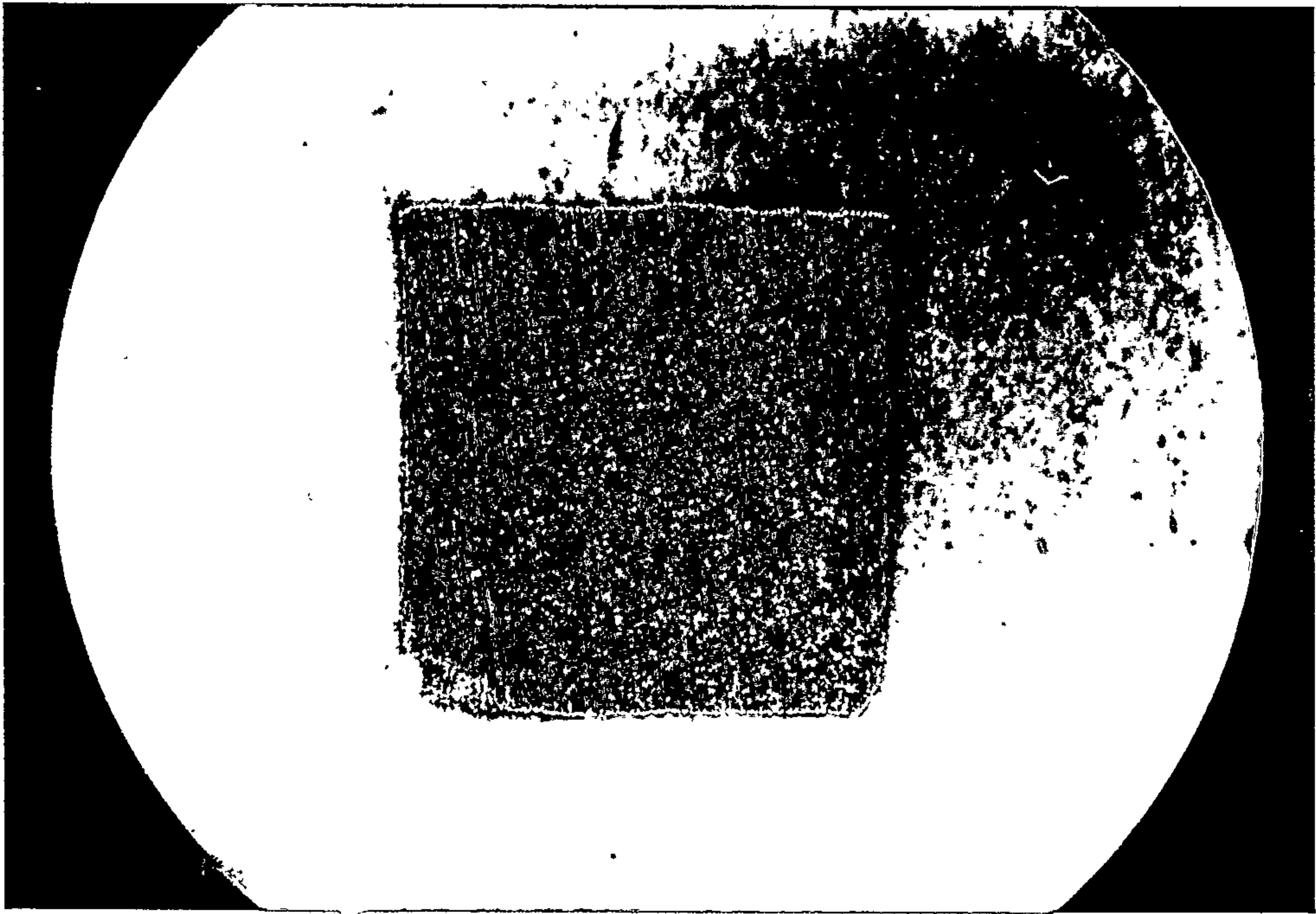


**Figure 18** Mounted gold surface under the Photomakroskop at 6.3 times magnification showing regular surface abrasions.

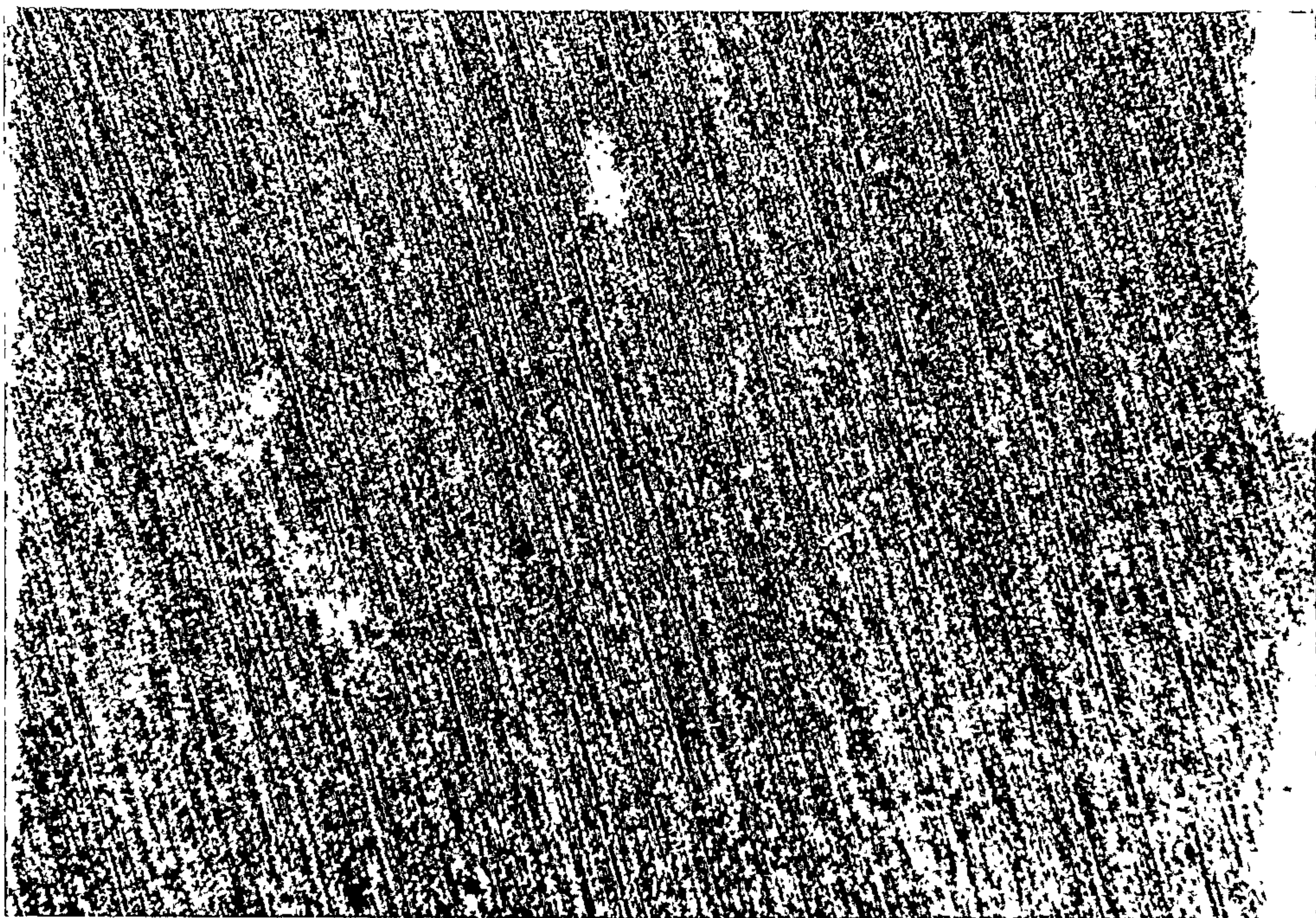


**Figure 19** Higher magnification (24 times) of Figure 18 showing uniformity of surface abrasion.

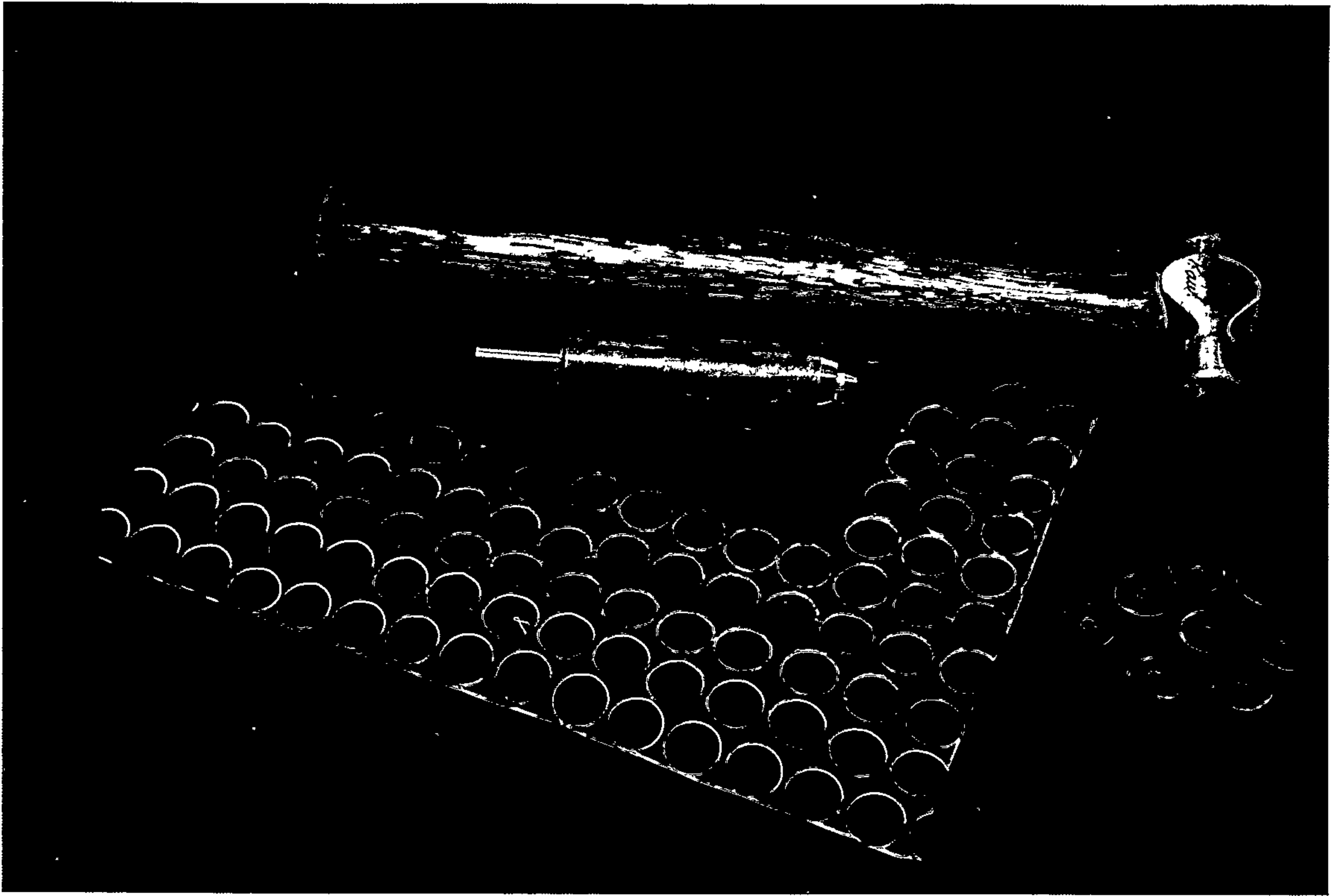


**Figure 20** Contact adhesive and punch used to construction adhesive isolating rings.

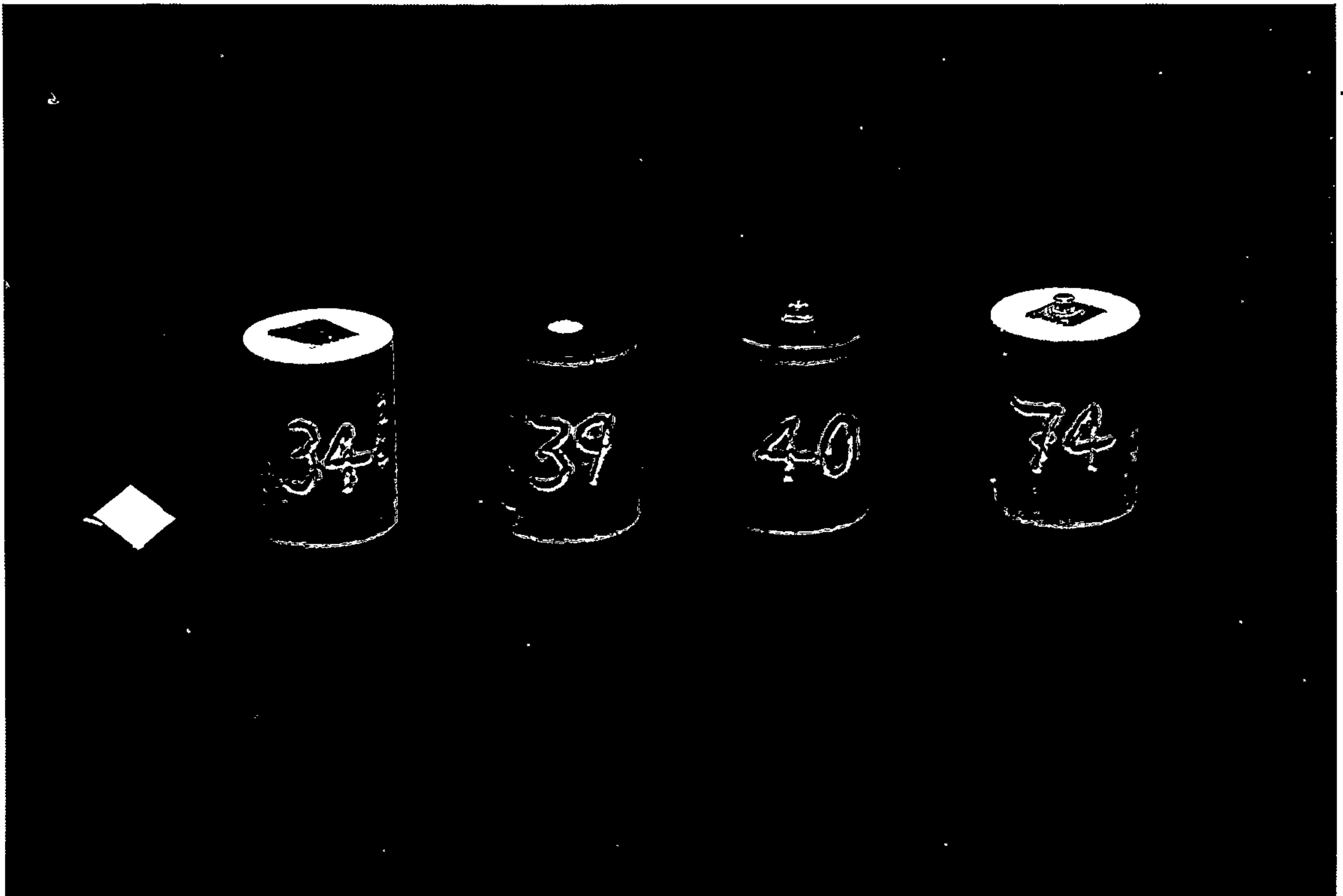


**Figure 21** Stages in the production of specimens prior to shear testing in the Hounsfield tensometer.

2) Thorough rinsing of the gold alloy surface with an air/water syringe to remove debris.

3) Drying thoroughly with moisture- and oil-free compressed air.

4) Applying 3M etching gel, provided with the Silar kit, to the alloy surface for 60 seconds.

5) Rinsing the specimen with a copious amount of water from the air/water syringe for 45 seconds.

6) Air drying with the air/water syringe.

7) Drying the alloy thoroughly with warm air from a hair dryer (Weisser 1973; Slodov & Armbrecht 1974; Reynolds 1975; Gorelick, Masunga, Thomas & Zachrisson 1978).

8) Mixing the Scotchbond liquid and resin together for 10 seconds.

9) Applying Scotchbond to both the alloy and the mesh surface of the lingual button with a soft brush.

10) Air drying both surfaces with the air/water syringe to leave a shiny film on the surface (Buonocore 1981a).

11) Reapplying the Scotchbond to the alloy surface and drying as before.

12) Mixing an equal amount of Paste A and B of self-curing Silar for 20 seconds. To obtain equal quantities of the two pastes they were first loaded into disposable syringes and equal lengths were expressed.

13) Applying an excessive amount of Silar to the mesh surface of the lingual button as it is held in bonding tweezers.

14) Placing the button over the circumscribed area on the alloy surface.

15) Seating the button with a 200 gram force in a specially constructed weight (see Figure 22) to control resin thickness (Low, Davies & von Fraunhofer 1975b; Hormati, Fuller & Denehy 1980; Mizrahi, Cleaton-Jones & Austin 1981) and prevent disturbance of the button during the critical setting reaction of the resin. Creugers (1986) notes that load application during setting also acts to decrease some of the setting shrinkage.

16) Removing excess resin from around the button, prior to polymerization, with a no. 6 probe (Jassem, Retief & Jamison 1981; Buzzitta, Hallgren & Powers 1982)

17) Waiting a minimum 2 1/2 minutes before removing the isolating adhesive by sectioning with a no. 11 scalpel blade. In effect, the adhesive was removed immediately prior to testing.

In a pilot study prior to the main experiment, it was determined that one factor producing variability was the method of removal of the isolating adhesive masking ring. If this ring was removed by simply lifting it at one corner, this appeared to stress the composite bond and create greater diversity in the bond testing results. There appears to be less variability in the results if the adhesive is cut from the gold in sections with a scalpel blade.

For Scotchprime a similar procedure was adopted:-

1) As for steps 1) to 7) for Scotchbond.

2) Apply a moderate amount of Scotchprime to the alloy surface.

3) Gently air dry the alloy surface with the air/water syringe for 10 seconds to evaporate the solvent and leave a dry surface.

4) As for steps 8) to 17) for Scotchbond.

For Fusion the procedure varied slightly:-

- 1) Equal proportions of Fusion liquids A and B were mixed together by thoroughly hand-shaking in the mixing jar provided, and allowed to stand for 15 minutes prior to use.
- 2) Alloy surfaces were cleaned with alcohol and air-dried with the air/water syringe.
- 3) Fusion was applied to the alloy and to the mesh surface of lingual button.
- 4) Specimens were allowed to stand undisturbed for 3 minutes.
- 5) The alloy and button were gently air dried with the air/water syringe to evaporate the solvent.
- 6) Follow steps 8) to 17) for Scotchbond.

For Panavia Ex the following procedure was employed:-

- 1) As for steps 1) to 3) for Scotchbond.
- 2) Apply etch L, provided in the Panavia kit, for 60 seconds.
- 3) Copiously rinse for 20 seconds with water from the air/water syringe.
- 4) Dry with air/water syringe.
- 5) Thoroughly dry with warm air from a hair dryer.
- 6) Dispense correct amounts of Panavia Ex powder and liquid using measuring tool provided.
- 7) Mix powder and liquid for 60 seconds.
- 8) Apply excessive amount of Panavia to mesh backing of lingual button.
- 9) Follow steps 14) to 16) for Scotchbond.
- 10) Apply Oxyguard provided in Panavia kit around circumference of button since resin in contact with air fails to

polymerize due to oxygen inhibition (Ceen & Gwinnett 1980; Pegoraro & Barrack 1987).

11) Wait 6 minutes.

12) Flush off Oxyguard with water from air/water syringe.

13) Remove isolating adhesive from alloy by sectioning with a no. 11 scalpel blade.

#### **12.4. SPECIMEN STORAGE**

The 10 minute specimens were kept dry prior to testing, whereas the 24 hour specimens were stored in filtered water in an incubator (Knoll, Gwinnett & Wolff 1986) at  $37^{\circ} \pm 2^{\circ}$  C after standing for 10 minutes. The 24 hour specimens were only removed from water immediately prior to testing.

#### **12.5. SHEAR TESTING**

The device used for testing was the Hounsfield Tensometer, Type W, motor driven, serial number W5617, used in its compression mode (see Figure 23) with a custom-made jig (see Figure 24) to allow shear stressing at a cross-head speed of 1/8th of an inch per minute. The 62 1/2 pound beam was employed with the appropriate scale and a new free-sliding pressure indicator. Prior to each specimen being tested the scale was re-calibrated to prevent any error in measurement. Adjustment was also made to ensure that the shearing load would be applied parallel (see Figure 25) to the bracket/gold interface (Alexandre, Young, Sandrik & Bowman 1981). Specimens were stressed to failure and the results recorded for statistical analysis. Following testing, specimens were re-examined under the Photomakroskop for failure sites at magnifications up to 36

times to determine whether failure was primarily cohesive or adhesive (Alexandre, Young, Sandrik & Bowman 1981) (see Figure 26).

## **6. SCANNING ELECTRON MICROSCOPY**

A selection of shear tested specimens and lingual buttons were also prepared for examination under a Phillips 505 scanning electron microscope (SEM) (see Figure 27) and a surface analysis performed utilising the EDAX (energy dispersive x-ray analysis) system. Specimens chosen were those exhibiting the higher test values which were deemed to be more likely to have formed an adhesive bond (Retief 1974). All specimens were carbon-coated prior to SEM examination and EDAX.

Micrographs of the representative surfaces were taken at varying magnifications to determine the extent of the gold bonding. Areas suspected of exhibiting adhesive remnants were subjected to EDAX and the results printed by a Hewlett-Packard plotter. Examination was also performed on the bonding surface of the corresponding lingual buttons.

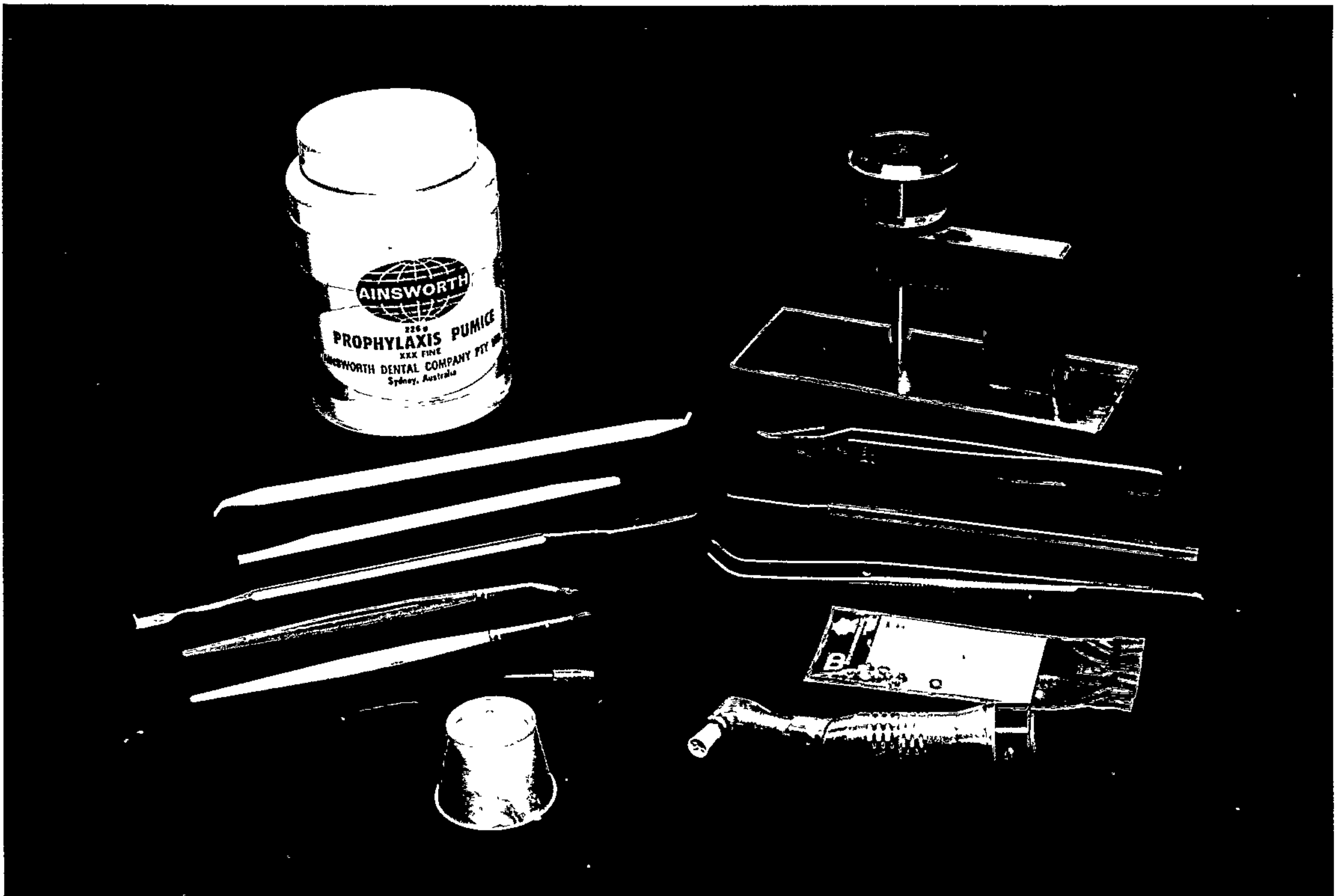


Figure 22 Instruments used to bond lingual buttons to gold alloy surfaces.

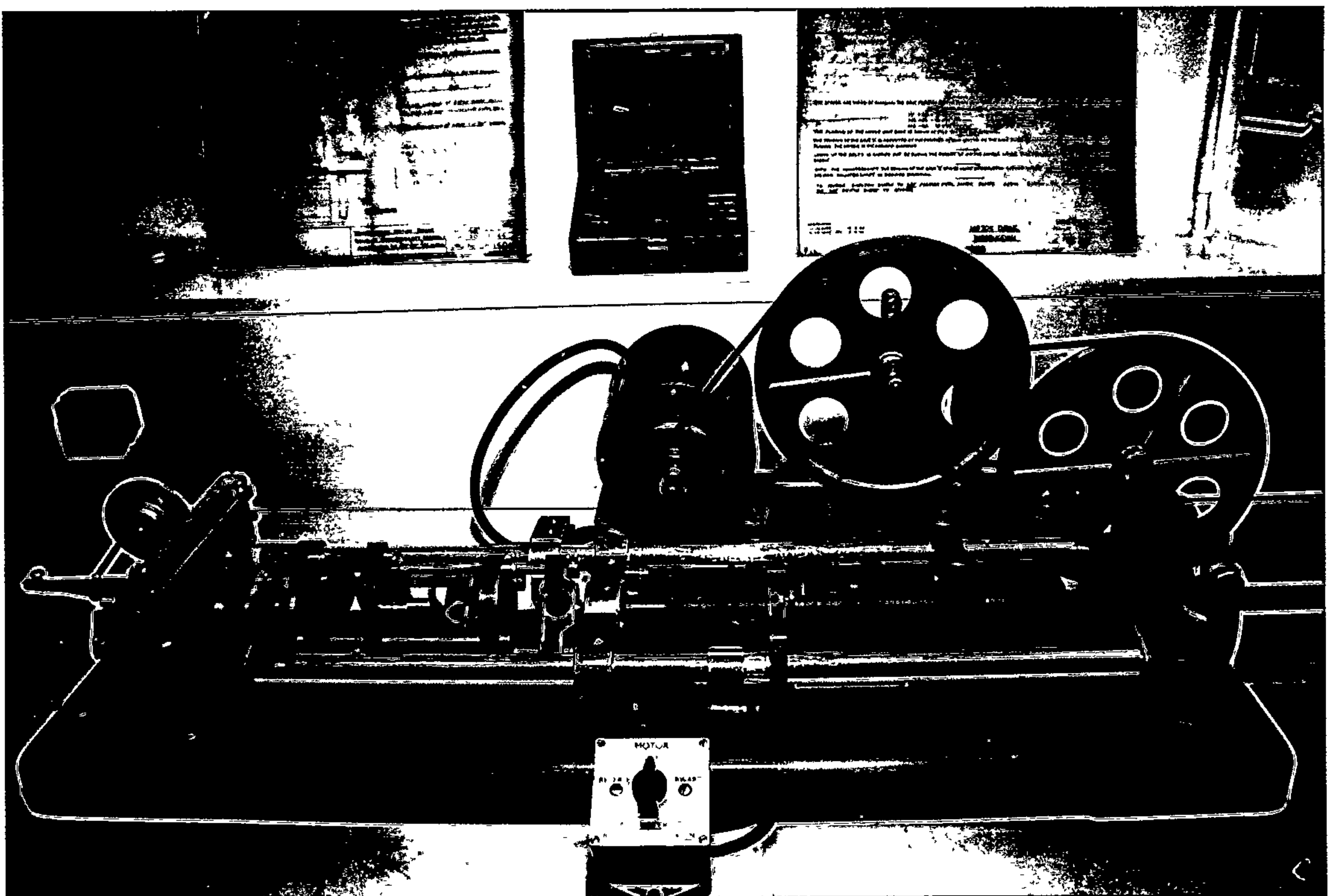


Figure 23 The Hounsfield Tensometer, serial number W5617, used to shear test the adhesives in this experiment.

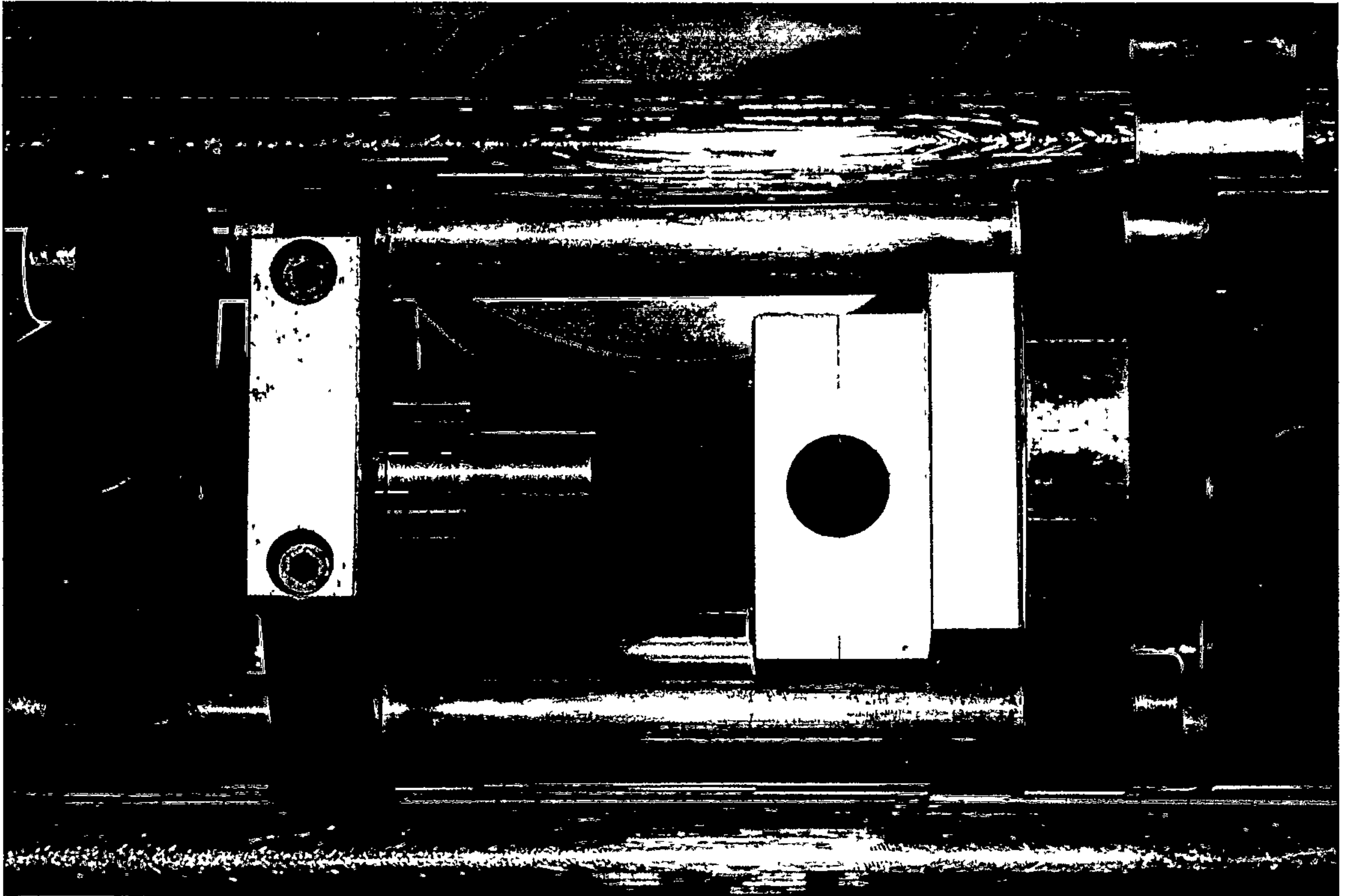


**Figure 24** Custom-built jig that permits mounting of specimens in the tensometer for shear testing.

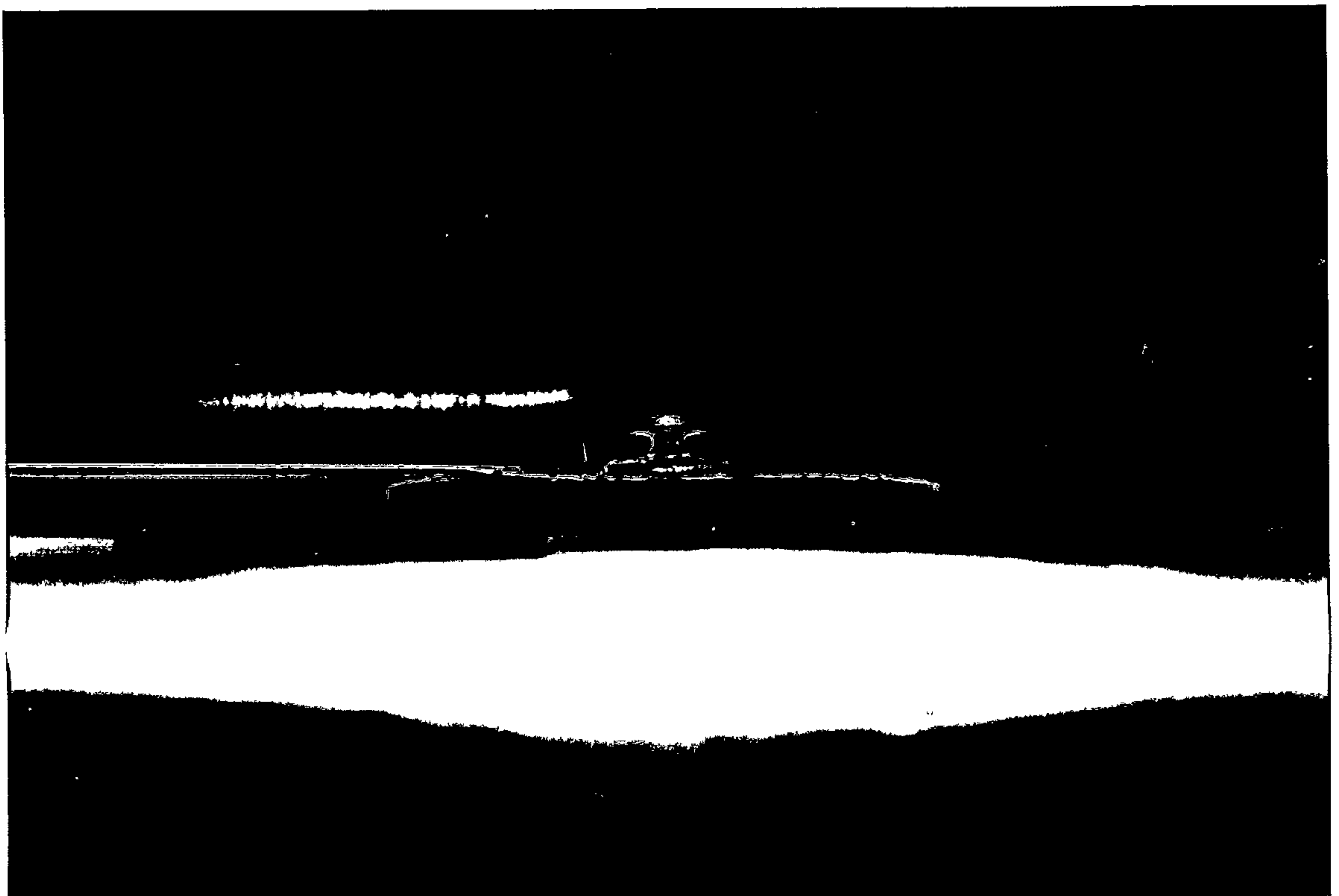


**Figure 25** Close-up photograph of shearing knife acting parallel to specimen surface.

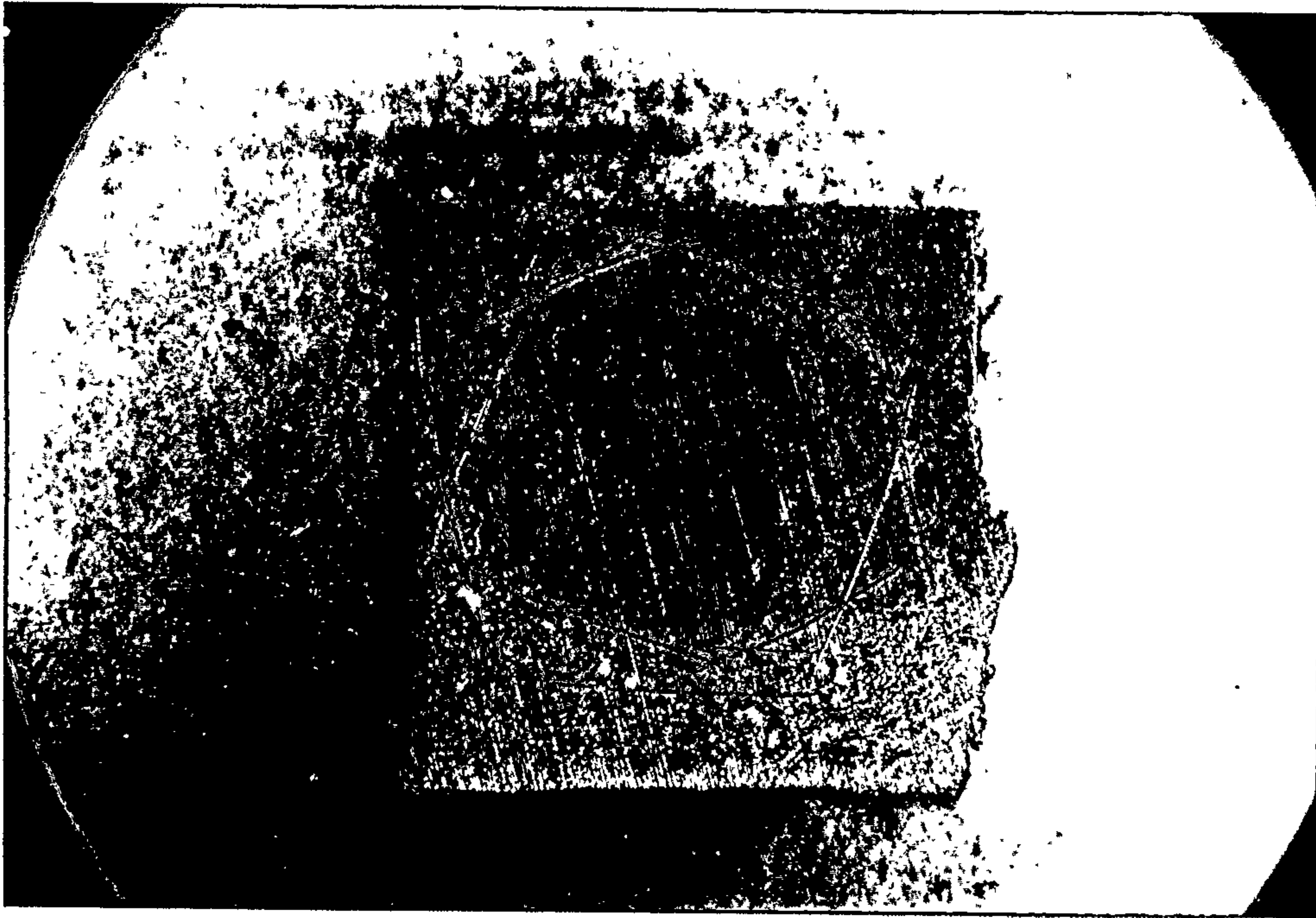


**Figure 26** Gold alloy surface following shear testing viewed under Photomakroskop at 6.3 times magnification.

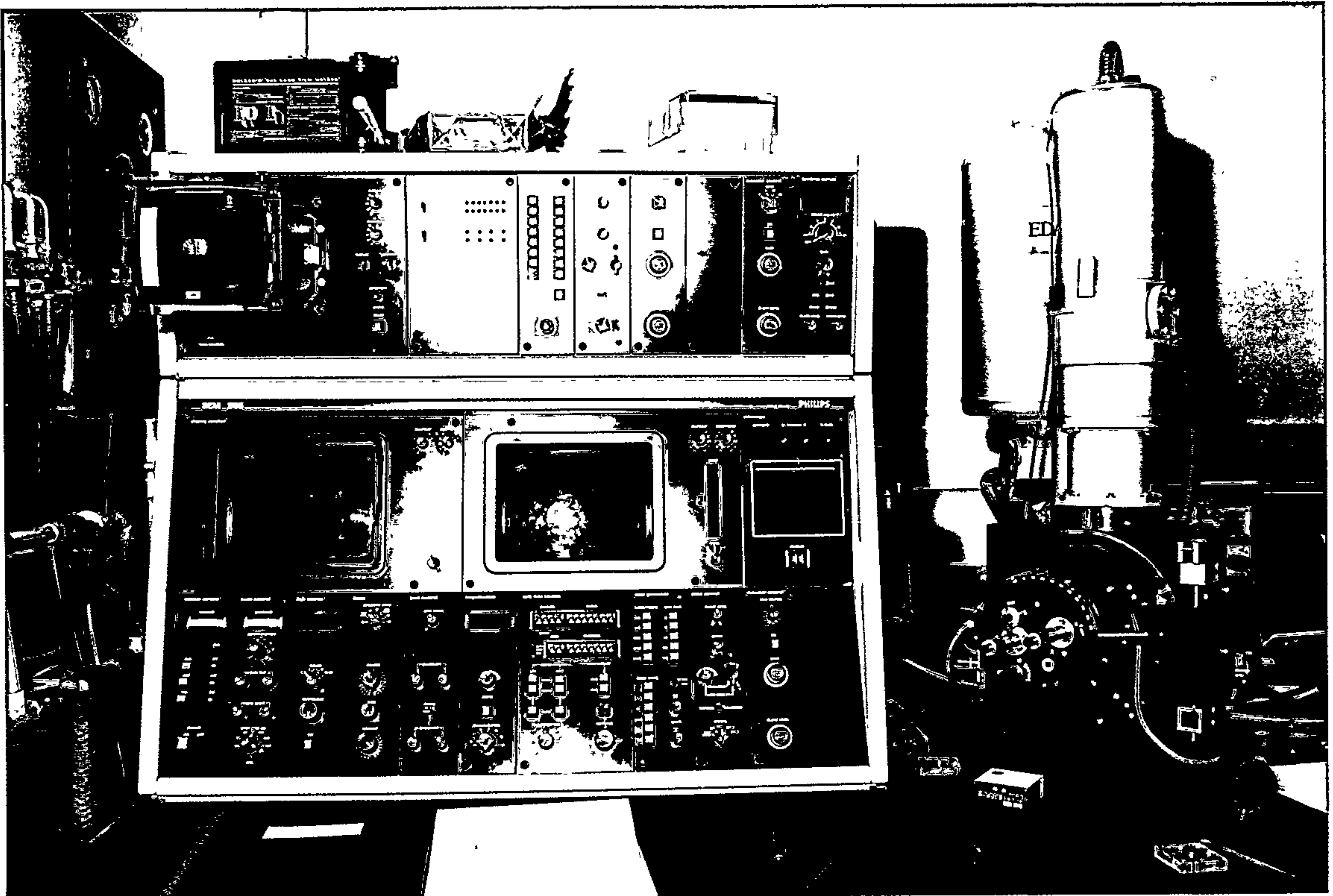
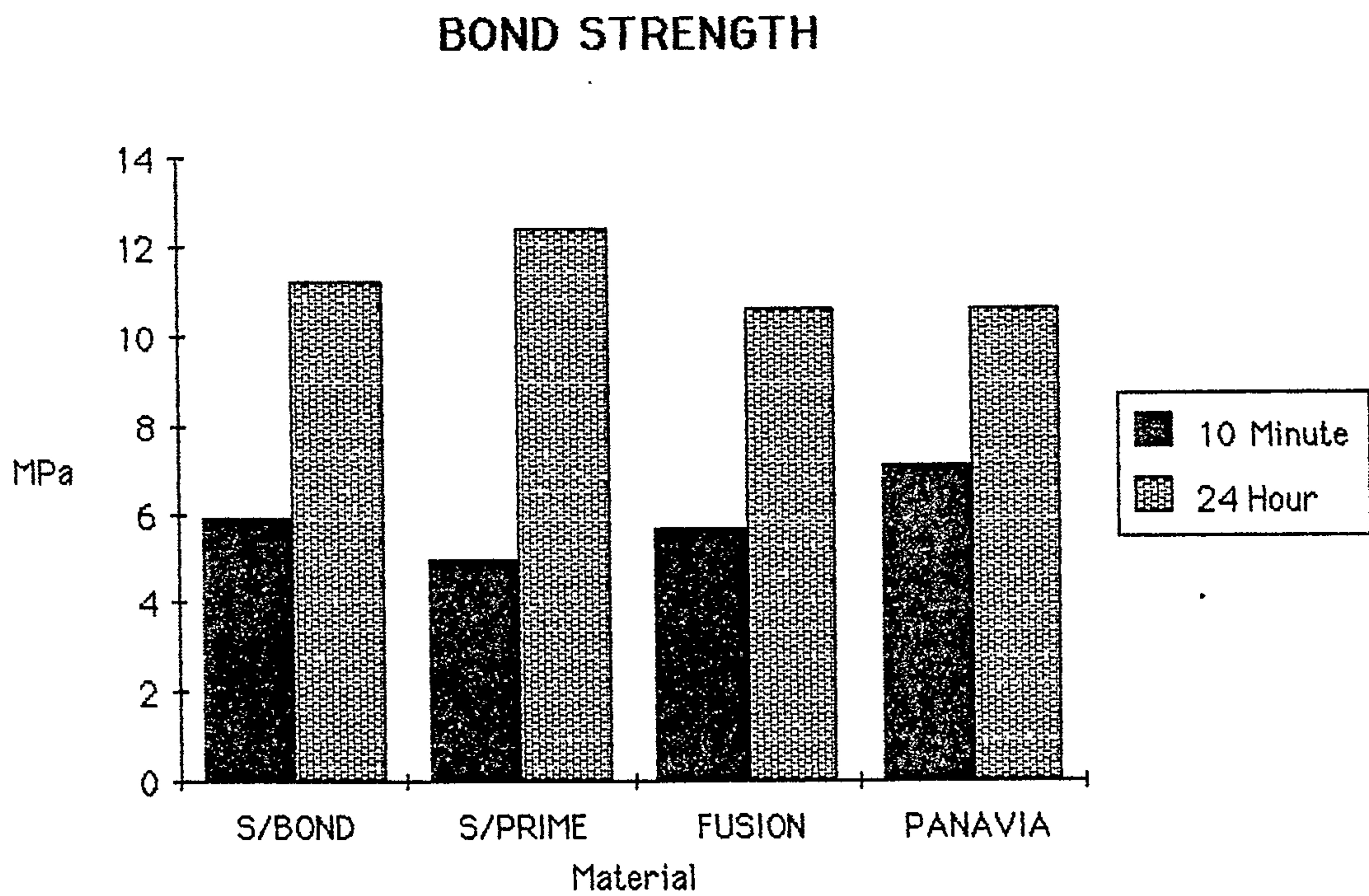


**Figure 27** Phillips 505 scanning electron microscope in the Electron Microscope Unit at the University of Sydney.

**Figure 28** Column graph of mean shear bond strengths at 10 minutes and 24 hours for all adhesives tested. Measurements converted to MPa.



## 13. RESULTS

### **13.1. BOND TESTS**

The numeric results for this study are presented in Figure 28 and Table 1. The co-efficient of variation for the results of the 10 minute test was 28%, and for the 24 hour test was 31%. Although these figures appear high, they are comparable to values presented by other authors (see Discussion).

A two-factor, factorial design, analysis of variance was used to statistically evaluate the experimental data. The F-ratios of the analysis were used to test for significant effects caused by adhesives, time, and interactions between adhesives and time (see Table 2).

Bond strength was always significantly greater after 24 hours than after 10 minutes ( $P < 0.01$ ), regardless of the adhesive being tested. The results, however, also revealed that there was no significant difference between the shear strengths of the four bonding agents tested, nor was there any significant interaction between the bonding agents and the time period. This implies that these two factors are independent of each other.

If, however, a one-way analysis of variance for shear strength is performed for both 10 minutes and 24 hours (see Table 3), a slightly different picture emerges. At 10 minutes there is a significant difference between the adhesives ( $P = 0.05$ ). This is not reflected in the analysis at 24 hours. Therefore, it may be concluded that one (or more) of the adhesives has a greater initial bond strength, but that after 24 hours this superiority is not maintained. From inspection of the mean shear

bond strengths in Table 1 it would appear that this adhesive is Panavia Ex.

In order to verify which of the adhesives has this improved initial strength, two sample T-tests were performed between the 10 minute values, the 24 hour values, and the 10 minute and 24 hour values. These results are tabulated in Tables 4 to 6.

From the T-test both Scotchbond and Panavia Ex were found to have significantly greater bond strength at 10 minutes than Scotchprime. No other relationships showed significance.

In agreement with the 24 hour analysis of variance, there was no significant difference between adhesives at the 24 hour T-test. As would be expected all adhesives showed a significant increase in bond strength from 10 minutes to 24 hours, with Panavia Ex showing the least statistically significant increase.

### 13.2. FAILURE SITES

Visual inspection of all the debonded specimens by the author, immediately following shear testing, revealed the predominant site of failure as being at the adhesive/substrate interface. This may be represented as a percentage of the samples by;

|                                  |     |
|----------------------------------|-----|
| adhesive/substrate.....          | 93% |
| adhesive/substrate/cohesive..... | 7%  |
| bracket/adhesive.....            | 0%  |

Examination under the Photomakroskop revealed a higher percentage of failures as adhesive/substrate/cohesive failures than initially observed (see figures 29 & 30). Any specimen exhibiting adhesive remnants on the gold alloy surface at

magnification 32 times was deemed to have failed within the adhesive. Thus the revised site of failure percentages become;

|                                  |     |
|----------------------------------|-----|
| adhesive/substrate.....          | 66% |
| adhesive/substrate/cohesive..... | 34% |
| bracket/adhesive.....            | 0%  |

Examination of selected specimens representative of each of the adhesives by scanning electron microscopy (SEM), revealed the presence of inclusions on the surface of the debonded gold alloy (see figure 31). All specimens examined exhibited scattered, high molecular weight particles to some extent on the specimen surface. None of the specimens demonstrated a substantial coverage. The greatest number of these inclusions was found on the specimen representing Scotchprime at 24 hours (figure 33). This was a noticeable increase in comparison to Scotchprime at 10 minutes (figure 32).

Panavia Ex at both 10 minutes (figure 34) and 24 hours (figure 35) showed little difference in the number of surface inclusions and both specimens were only sparsely covered. Fusion at 10 minutes (figure 36) exhibited a higher proportion of inclusions than did Fusion at 24 hours (see Figure 37). Scotchbond at 10 minutes (figure 38), as would be expected, produced a very clean surface. However, at 24 hours (figure 39), it showed a greater number of inclusions than most of the other specimens, and was second only in coverage to Scotchprime at 24 hours.

### 13.3. SURFACE ANALYSIS

Analysis of the gold alloy surfaces with the EDAX system (figures 40 to 49) demonstrated the presence of silicon on all specimens, with the exception of Scotchbond at 10 minutes (figure 44). This verified the SEM observations and confirmed that the inclusions noted on the alloy surfaces were in fact remnants of the bonding agents used in the experiment.

Examination of the bonding surfaces of the corresponding lingual buttons produced, as expected, predominantly silicon (see Figure 50), with the exception of the button corresponding to Scotchprime at 24 hours which also showed traces of gold (see figure 40 & 49).

**Table I****RESULTS OF 10 MINUTE TEST (MPa)**

| Adhesive    | Sample<br>Size | Mean  | S.D.  | S.E.<br>Mean | Min.  | Max.   |
|-------------|----------------|-------|-------|--------------|-------|--------|
| Scotchbond  | 10             | 5.850 | 1.228 | 0.388        | 3.950 | 7.850  |
| Scotchprime | 10             | 4.954 | 0.550 | 0.174        | 4.360 | 6.130  |
| Fusion      | 10             | 5.684 | 1.766 | 0.559        | 3.840 | 8.940  |
| Panavia ex  | 10             | 7.066 | 2.386 | 0.754        | 3.430 | 10.910 |

**RESULTS OF 24 HOUR TEST (MPa)**

| Adhesive    | Sample<br>Size | Mean   | S.D.  | S.E.<br>Mean | Min. | Max.  |
|-------------|----------------|--------|-------|--------------|------|-------|
| Scotchbond  | 10             | 11.200 | 3.700 | 1.170        | 5.30 | 17.56 |
| Scotchprime | 10             | 12.374 | 2.779 | 0.879        | 8.94 | 16.42 |
| Fusion      | 10             | 10.639 | 2.732 | 0.864        | 7.38 | 14.86 |
| Panavia ex  | 10             | 10.630 | 4.310 | 1.360        | 4.68 | 18.49 |

All tables produced by computer generated statistics programmes. See Appendix C for references.

Table 2**ANALYSIS OF VARIANCE**

Two-factor, factorial treatment design

Factor 1 = Adhesive at 4 levels i.e. Scotchbond,  
Scotchprime, Fusion, Panavia ex.

Factor 2 = Time at 2 levels i.e. 10 min., 24 hrs.

Variate: Shear Strength (MPa)

| Source of variation | df | sums of<br>squares% | variance | F-ratio | P      |
|---------------------|----|---------------------|----------|---------|--------|
| Main Effects        |    |                     |          |         |        |
| Adhesive            | 3  | 0.45                | 1.687    | 0.232   | NS     |
| Time                | 1  | 50.02               | 566.367  | 78.027  | < 0.01 |
| 2-Way Interactions  |    |                     |          |         |        |
| Adhesive vs. Time   | 3  | 3.37                | 12.718   | 1.752   | NS     |
| Residual            | 72 | 46.16               | 7.259    |         |        |
| Total               | 79 | 100.00              |          |         |        |

Grand Mean                      8.55

**Table 3****ANALYSIS OF VARIANCE**

One-way analysis on shear strength after 10 minutes (MPa).

| Source of variation | df | sums of squares% | variance | F- ratio | P      |
|---------------------|----|------------------|----------|----------|--------|
| Adhesive            | 3  | 19.41            | 7.667    | 2.891    | = 0.05 |
| Residual            | 36 | 80.59            | 2.655    |          |        |
| Total               | 39 | 100.00           |          |          |        |

Grand Mean 5.89

**ANALYSIS OF VARIANCE**

One-way analysis on shear strength after 24 hours (MPa).

| Source of variation | df | sums of squares% | variance | F- ratio | P  |
|---------------------|----|------------------|----------|----------|----|
| Adhesive            | 3  | 4.51             | 6.73     | 0.567    | NS |
| Residual            | 36 | 95.49            | 11.86    |          |    |
| Total               | 39 | 100.00           |          |          |    |

Grand Mean 11.21

**Table 4****TWO-SAMPLE T-TEST**

Comparision between adhesive at 10 minutes and 24 hours.

| Adhesive    | T- value | P      | Comment            |
|-------------|----------|--------|--------------------|
| Scotchbond  | 4.34     | 0.0004 | highly significant |
| Scotchprime | 8.28     | 0.0000 | highly significant |
| Fusion      | 4.82     | 0.0000 | highly significant |
| Panavia ex  | 2.29     | 0.0350 | significant        |

where:  $df = 18$

$t_{18(.05)} = 2.101$

$t_{18(.01)} = 2.878$

Table 5**TWO-SAMPLE T- TEST**

Comparision between adhesives at 10 minutes

| Adhesives      | T- value | P     | Comment         |
|----------------|----------|-------|-----------------|
| Scotchbond vs  |          |       |                 |
| Scotchprime    | 2.11     | 0.049 | significant     |
| Fusion         | 0.24     | 0.810 | not significant |
| Panavia ex     | 1.43     | 0.170 | not significant |
| Scotchprime vs |          |       |                 |
| Fusion         | 1.25     | 0.230 | not significant |
| Panavia ex     | 2.73     | 0.014 | significant     |
| Fusion vs      |          |       |                 |
| Panavia ex     | 1.47     | 0.160 | not significant |

where:  $df = 18$ 

$$t_{18(.05)} = 2.101$$

$$t_{18(.01)} = 2.878$$

Table 6

**TWO-SAMPLE T- TEST**

Comparision between adhesives at 24 hours

| Adhesives      | T- value | P    | Comment         |
|----------------|----------|------|-----------------|
| Scotchbond vs  |          |      |                 |
| Scotchprime    | 0.80     | 0.43 | not significant |
| Fusion         | 0.38     | 0.71 | not significant |
| Panavia ex     | 0.32     | 0.76 | not significant |
| Scotchprime vs |          |      |                 |
| Fusion         | 1.41     | 0.18 | not significant |
| Panavia ex     | 1.08     | 0.30 | not significant |
| Fusion vs      |          |      |                 |
| Panavia ex     | 0.01     | 1.00 | not significant |

where:            df = 18  
                      $t_{18(.05)} = 2.101$   
                      $t_{18(.01)} = 2.878$

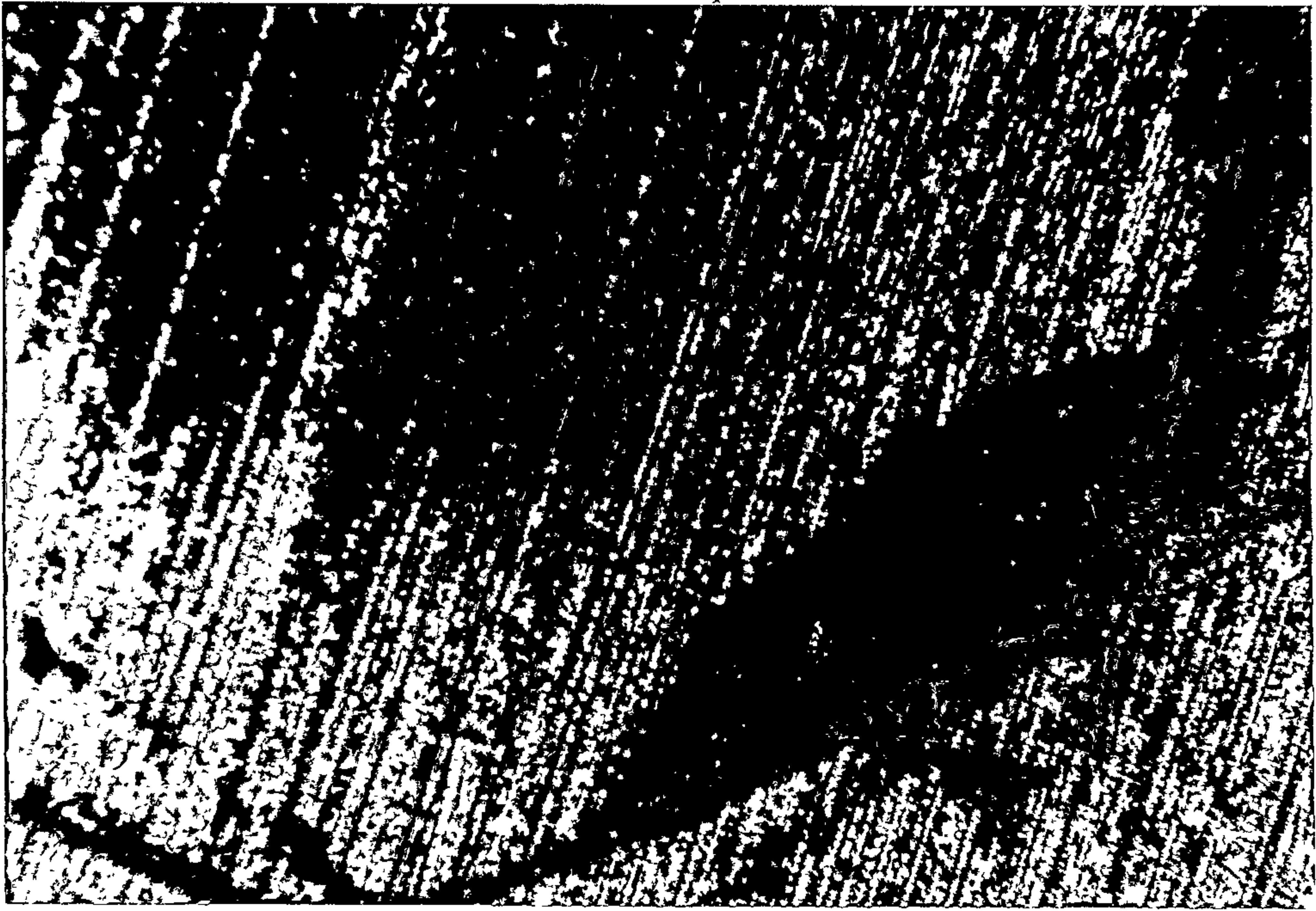


**Figure 29** Scotchprime 24 hour specimen examined under the Photomakroskop at 32 times magnification, with inclusions.

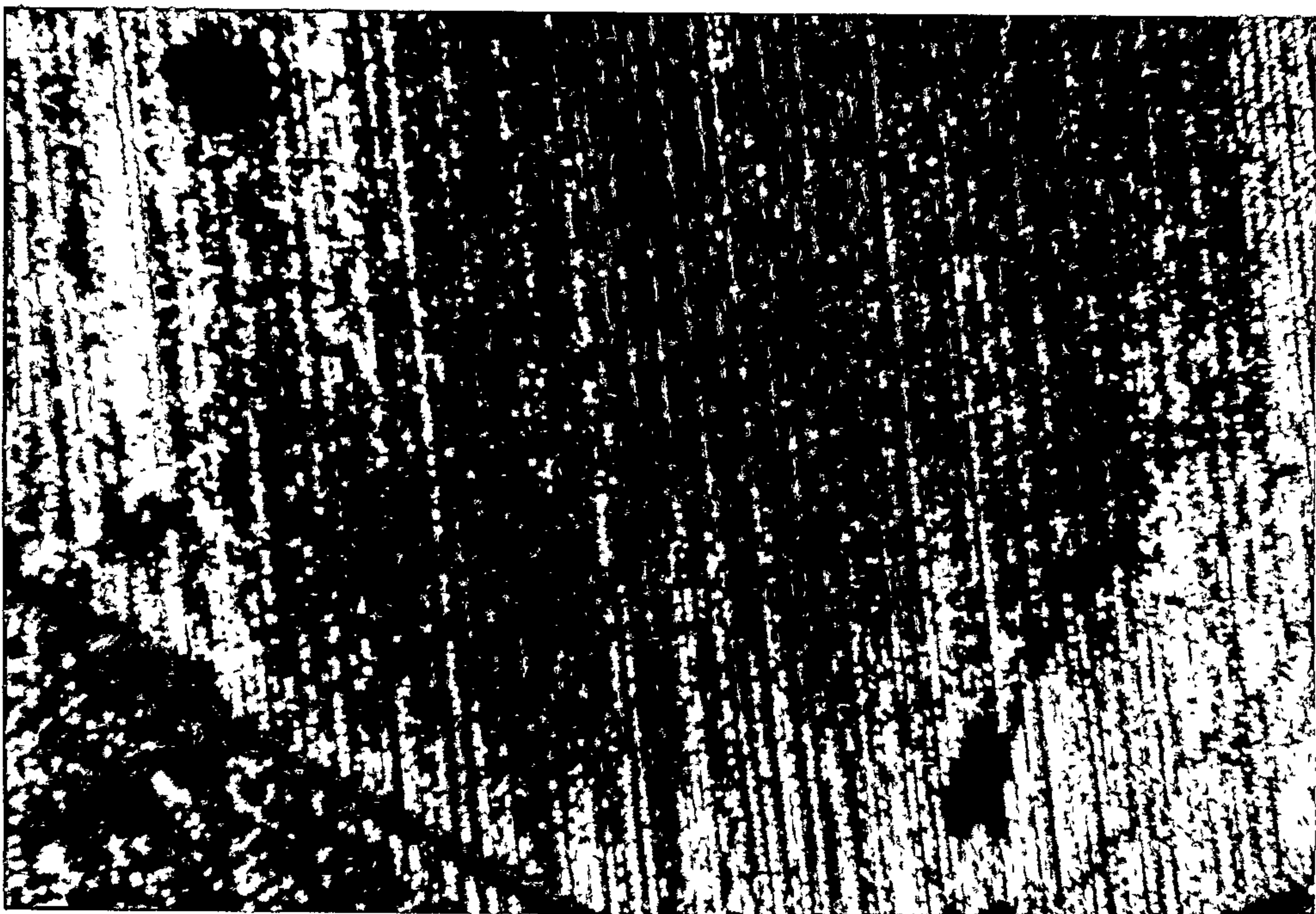


**Figure 30** Fusion 24 hour specimen, similar view to Figure 29 also showing inclusions, although reduced in size.

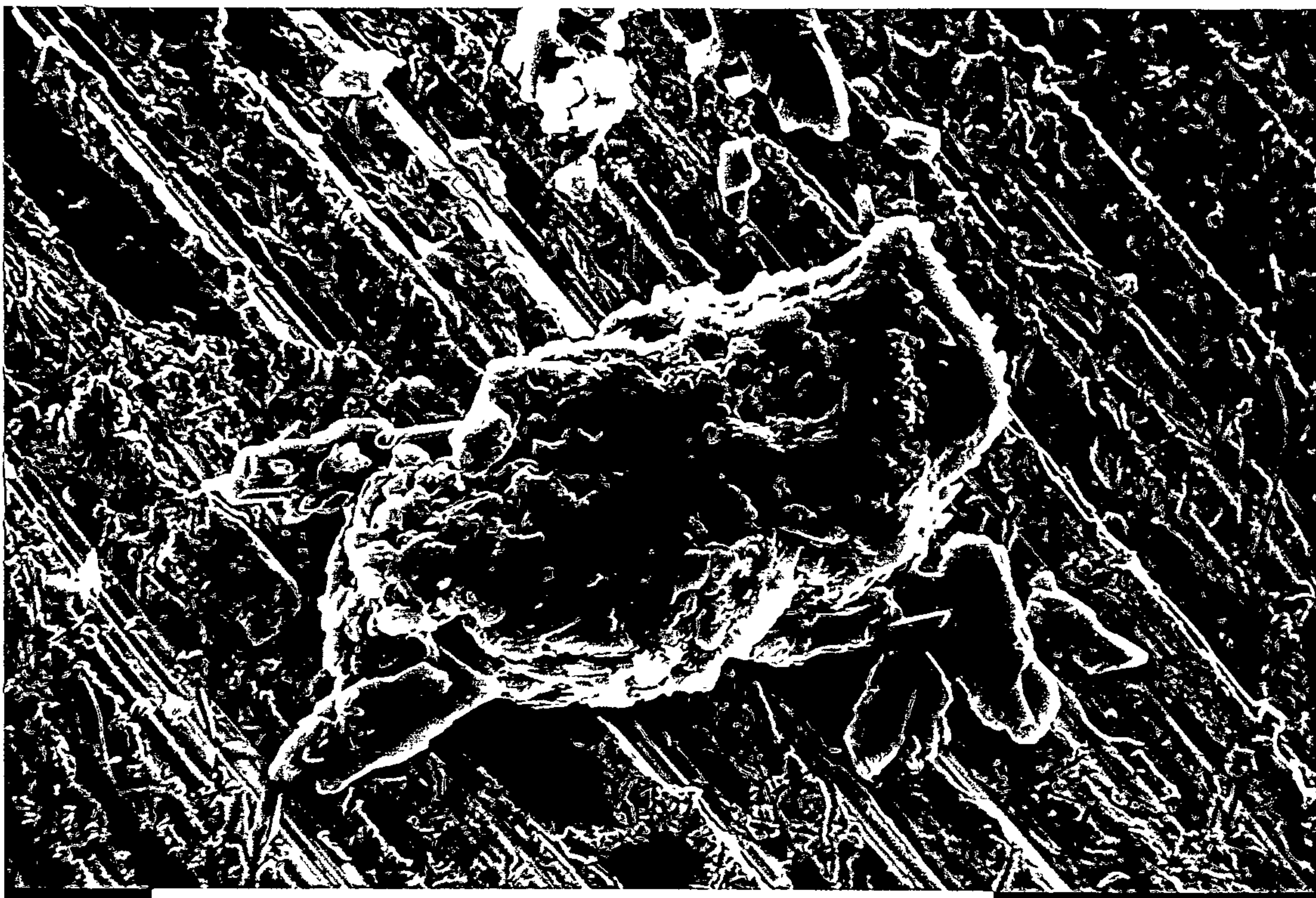


**Figure 31** Micrograph of inclusion on surface of Scotchprime 24 hour specimen magnified 775 times. Scale rep. 0.1mm.

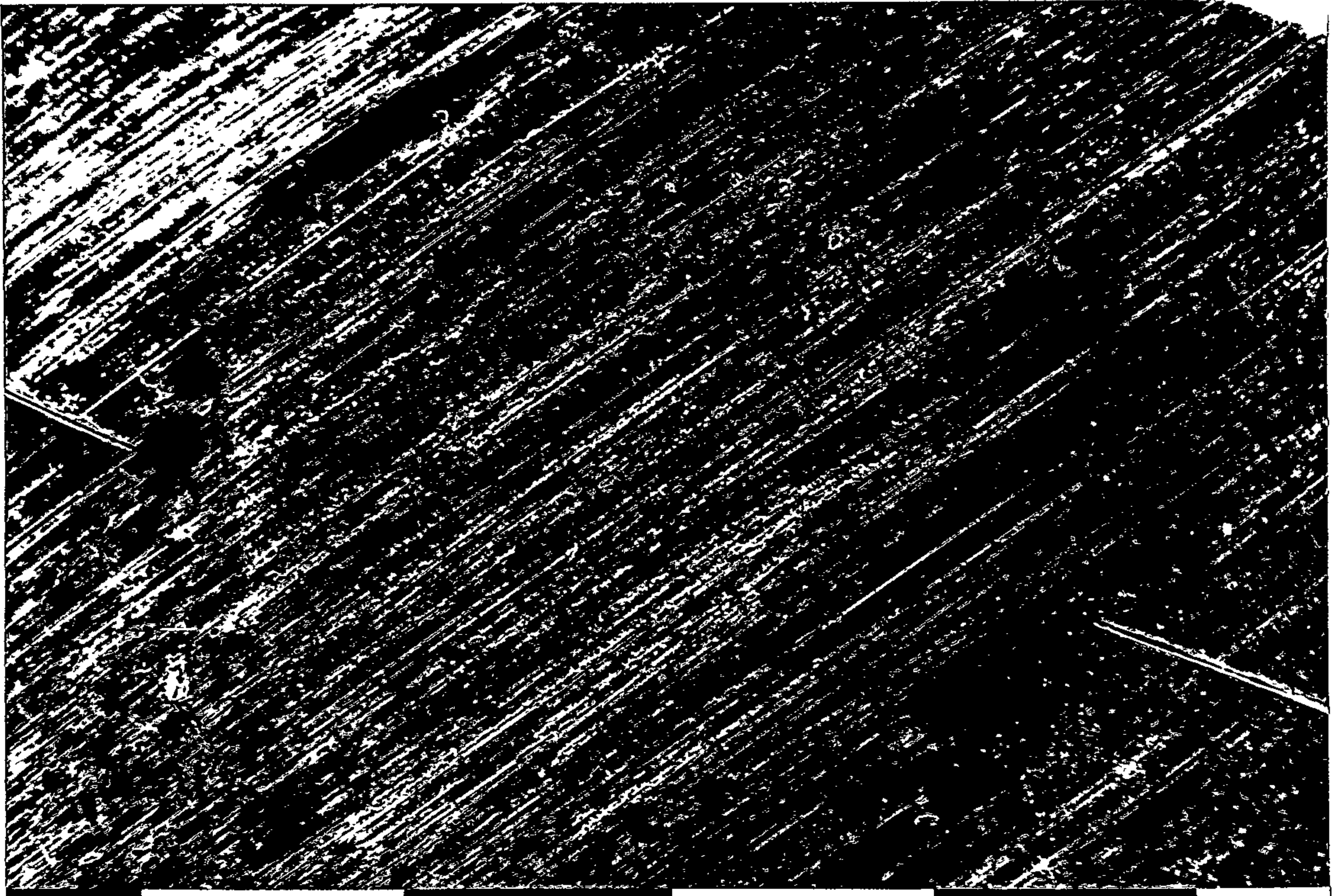


**Figure 32** Scotchprime 10 minute specimen showing scattered inclusions, magnified 242 times. Scale rep. 1.0mm.



**Figure 33** Scotchprime 24 hour specimen with noticeable inclusions magnified 242 times. Scale rep. 1.0mm.

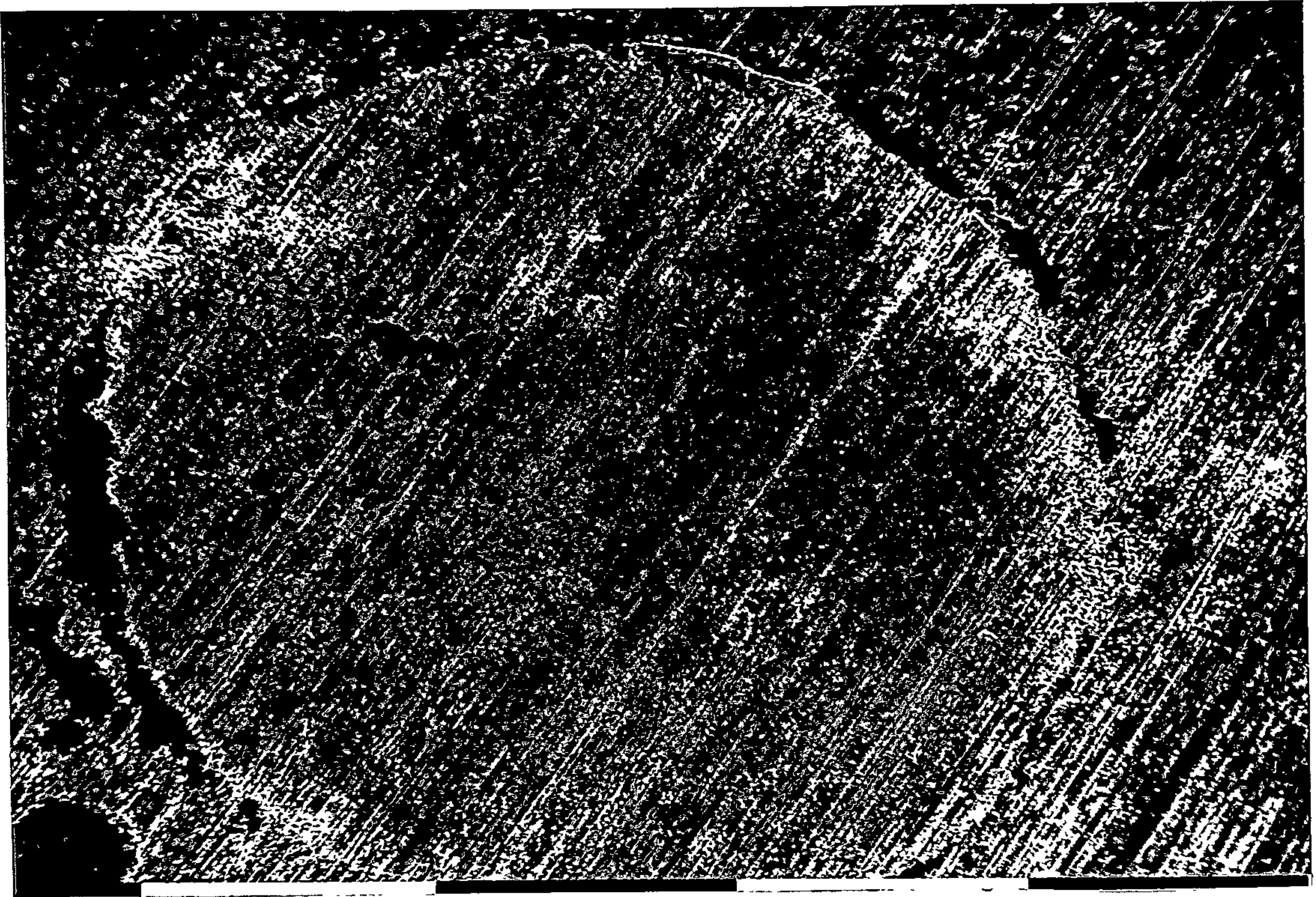


**Figure 34** Panavia Ex 10 minute specimen showing minimal inclusions, magnified 264 times. Scale rep. 1.0mm.

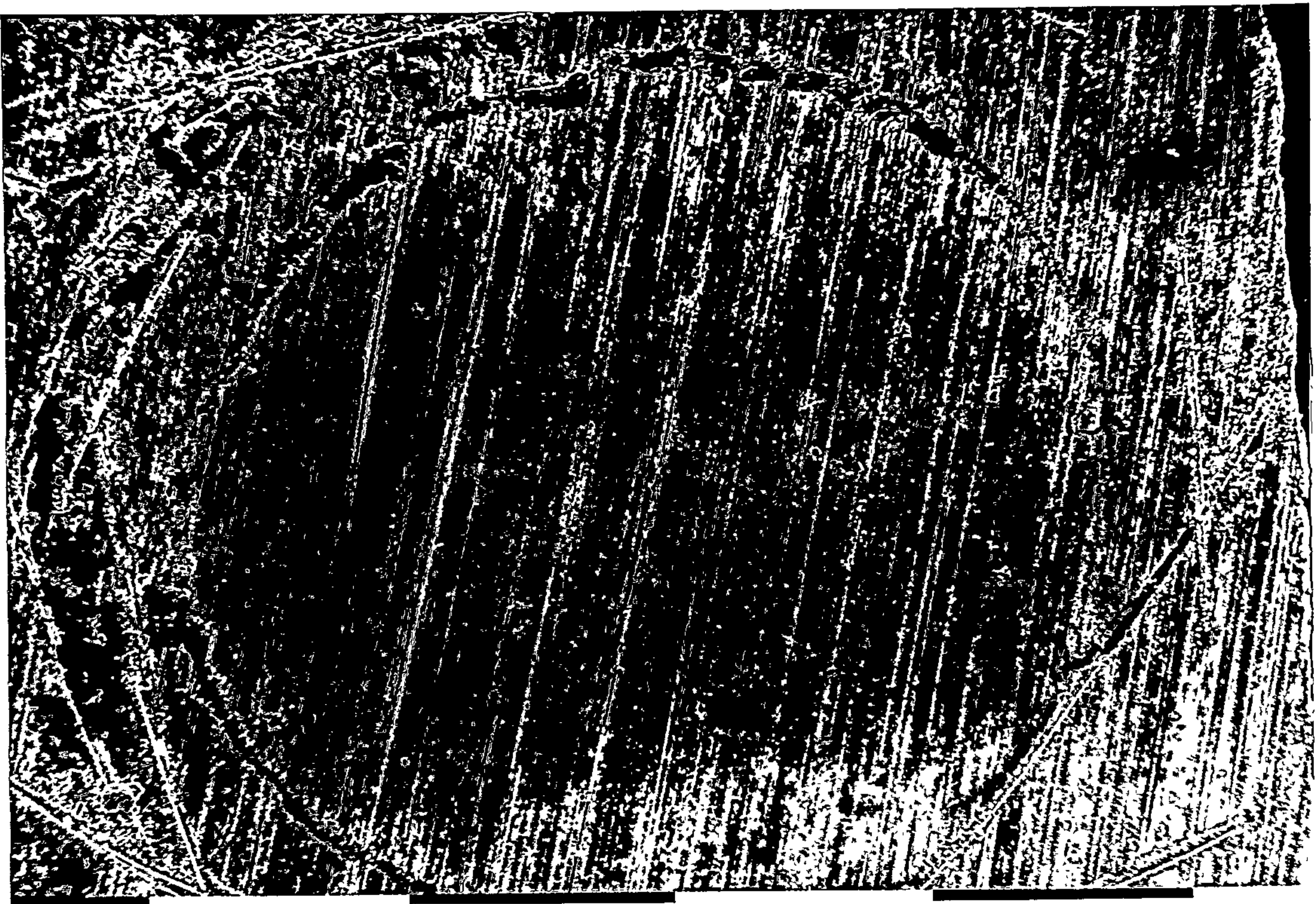


**Figure 35** Panavia Ex 24 hour specimen also with minimal inclusions, magnified 264 times. Scale rep. 1.0mm.

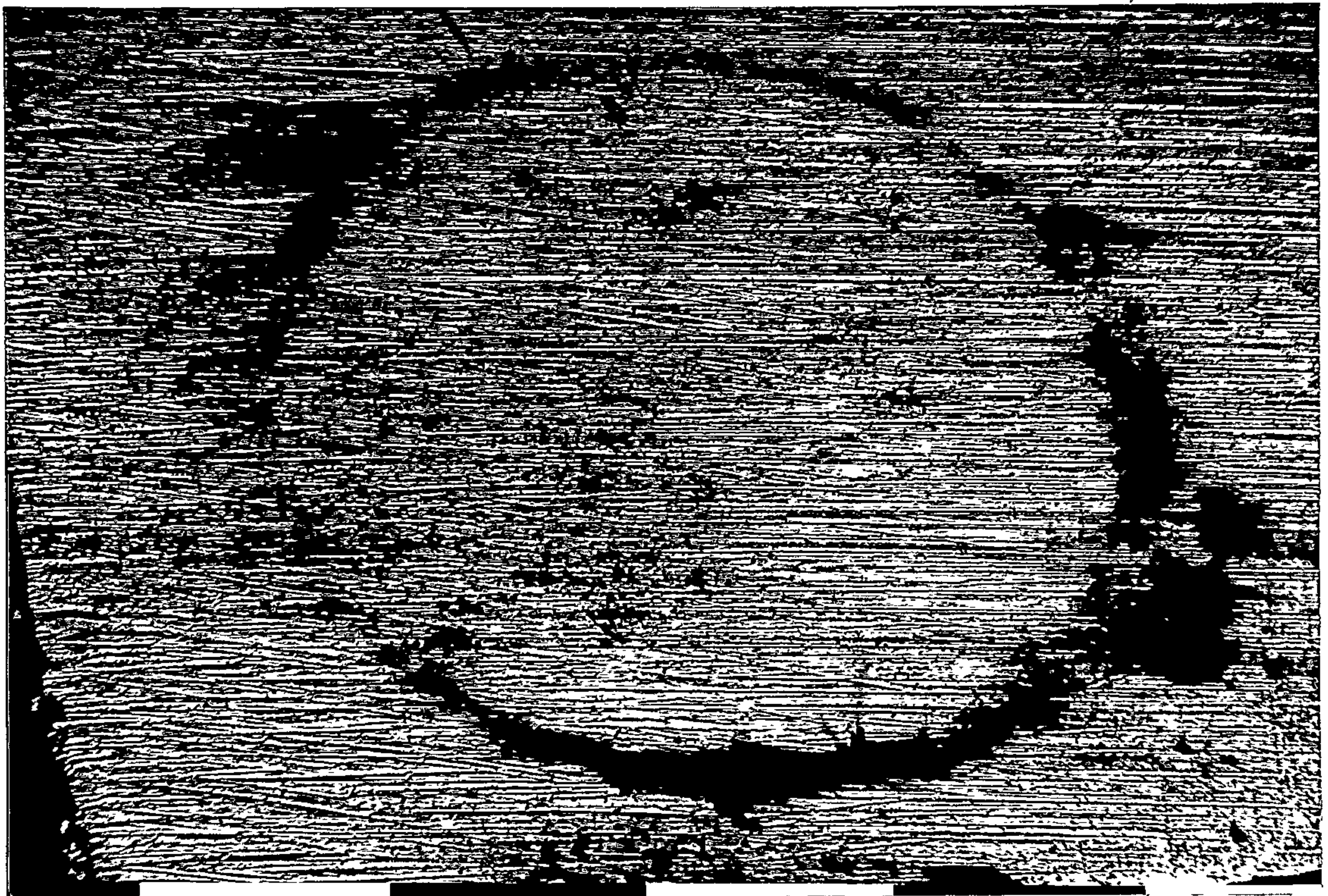


**Figure 36** Fusion 10 minute specimen with small scattered inclusions, magnified 230 times. Scale rep. 1.0mm.

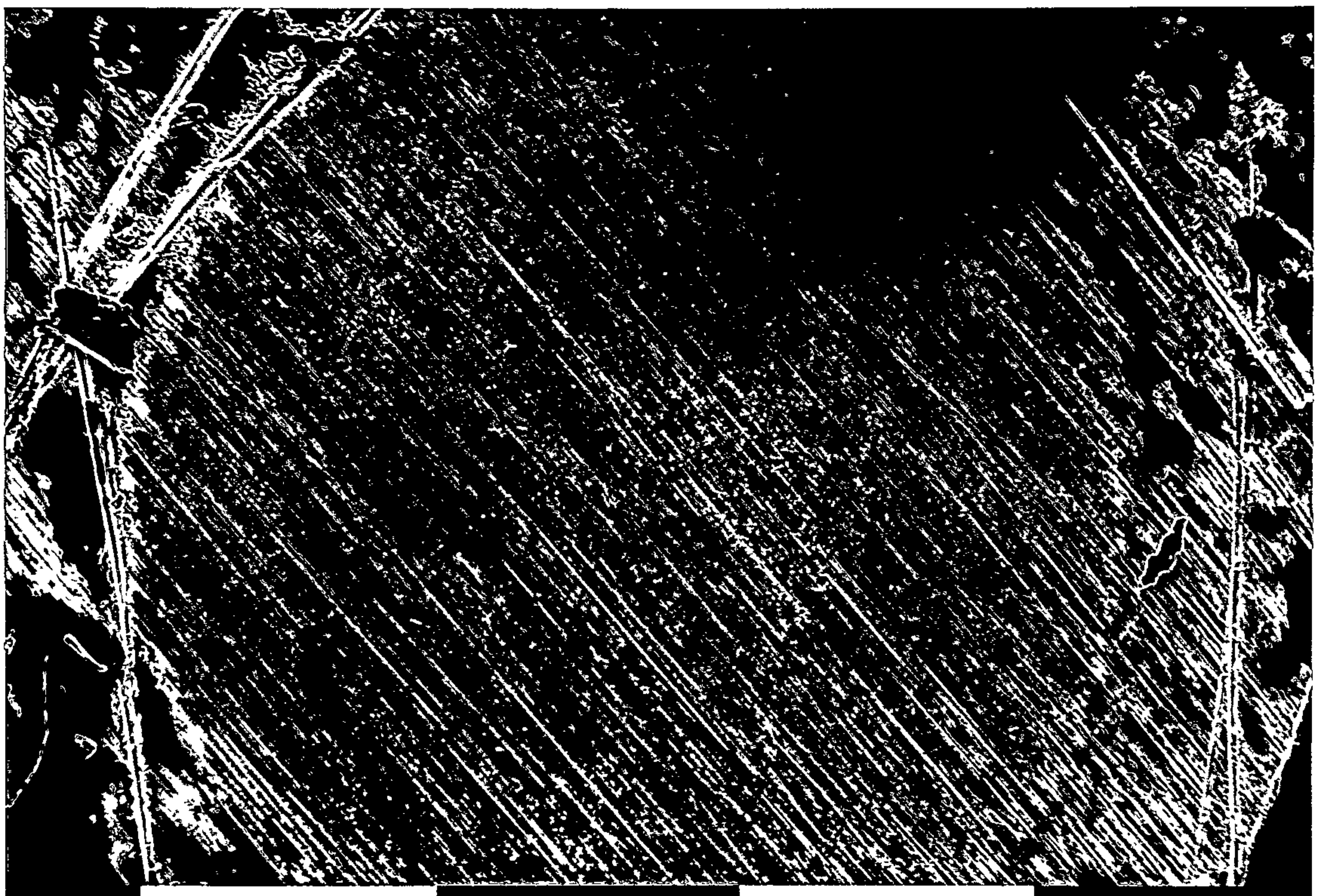


**Figure 37** Fusion 24 hour specimen with negligible evidence of inclusions, magnified 242 times. Scale rep. 1.0mm.



**Figure 38** Scotchbond 10 minute specimen with no evidence of inclusions, magnified 230 times. Scale rep. 1.0mm.

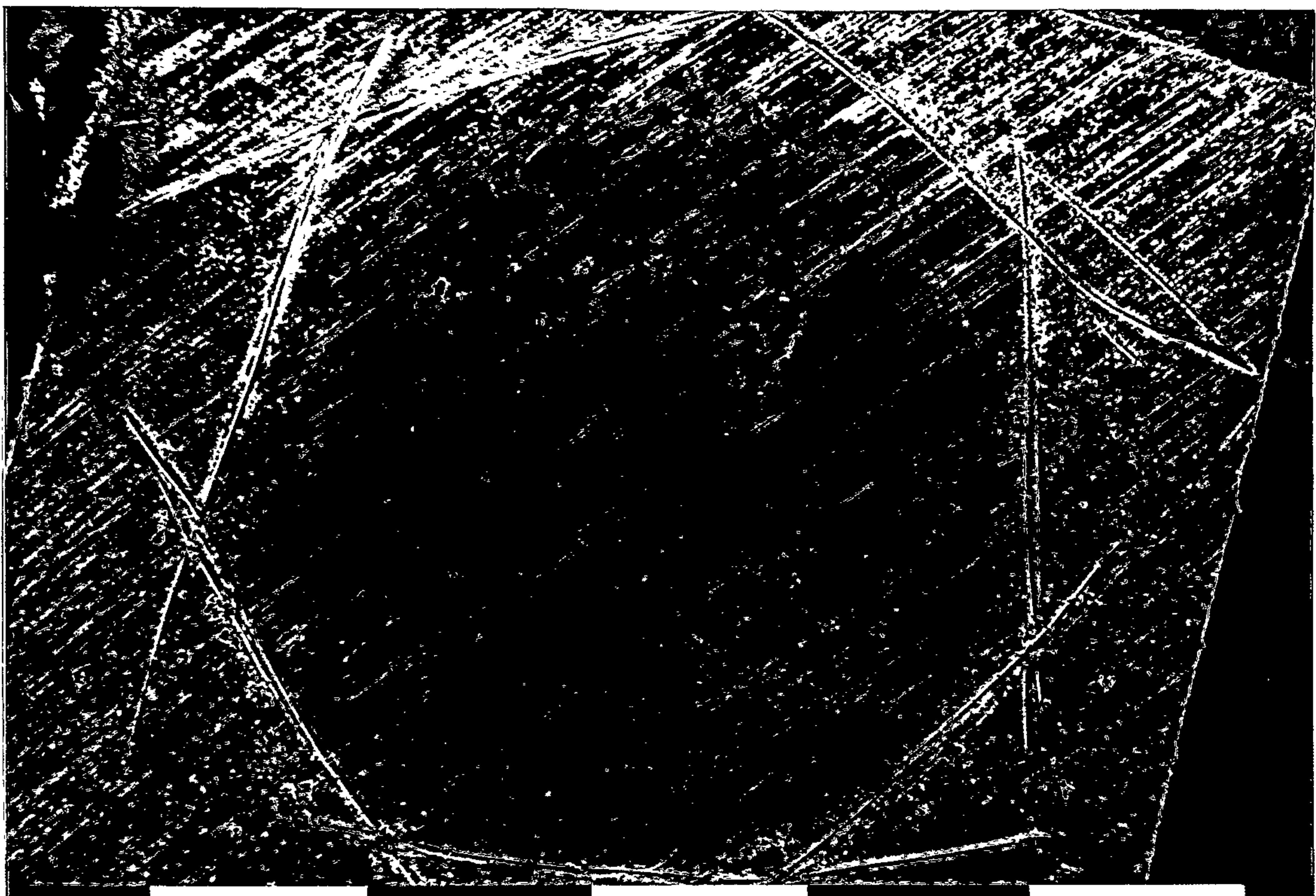


**Figure 39** Scotchbond 24 hour specimen with noticeable inclusions, magnified 230 times. Scale rep. 1.0mm.

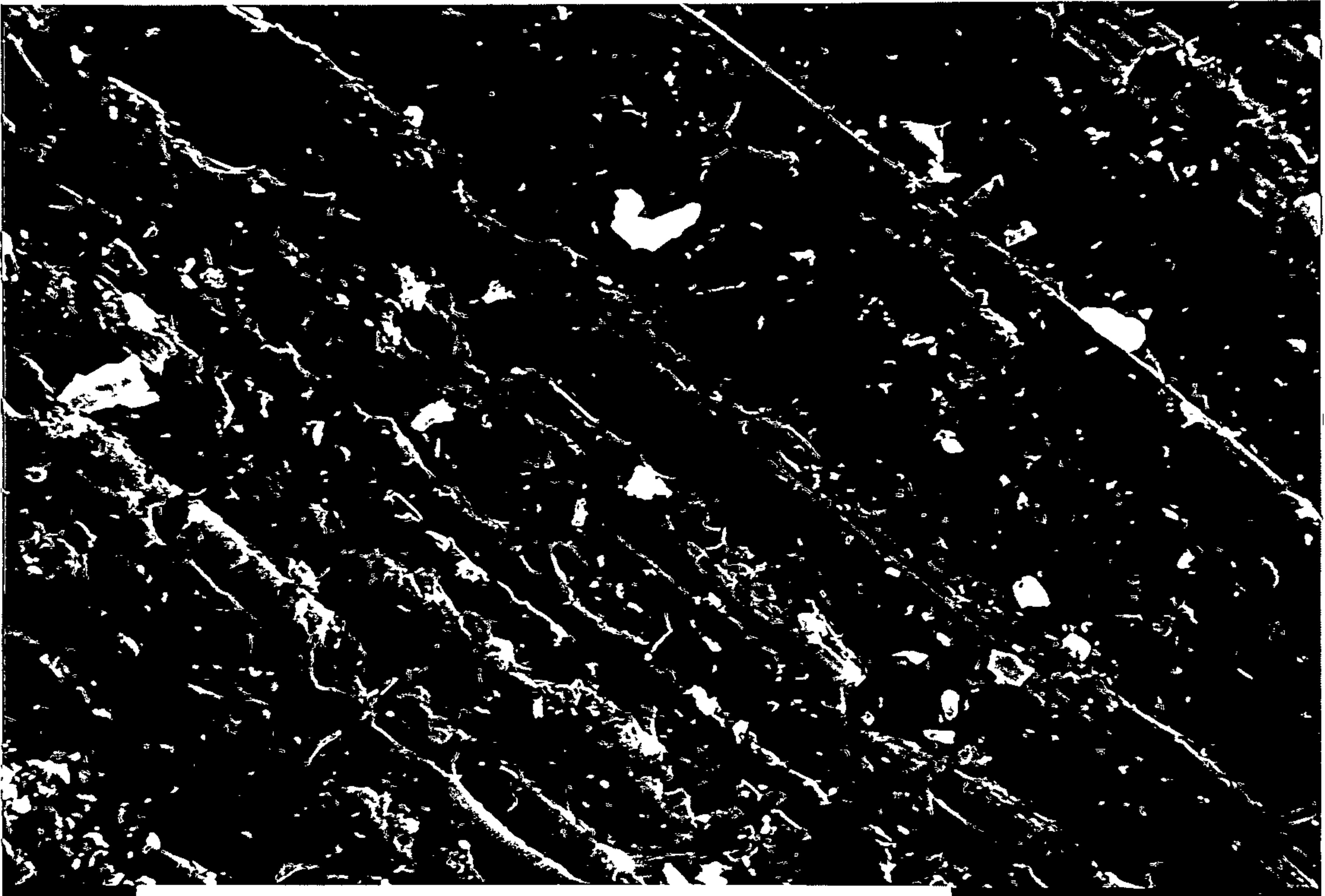


**Figure 40** Scotchprime 24 hour lingual button specimen, magnified 745 times, which exhibited traces of gold on EDAX. Scale rep. 0.1mm.

Figure 41 EDAX analysis of Fusion 10 minute specimen

18 - JUN - 87 16:21:30 EDAX READY  
 RATE = 10020 CPS TIME = 118 LSEC  
 FS = 2000 CNT PRST = DEF  
 B = INCLUSN PLATE SURFCE SPEC2

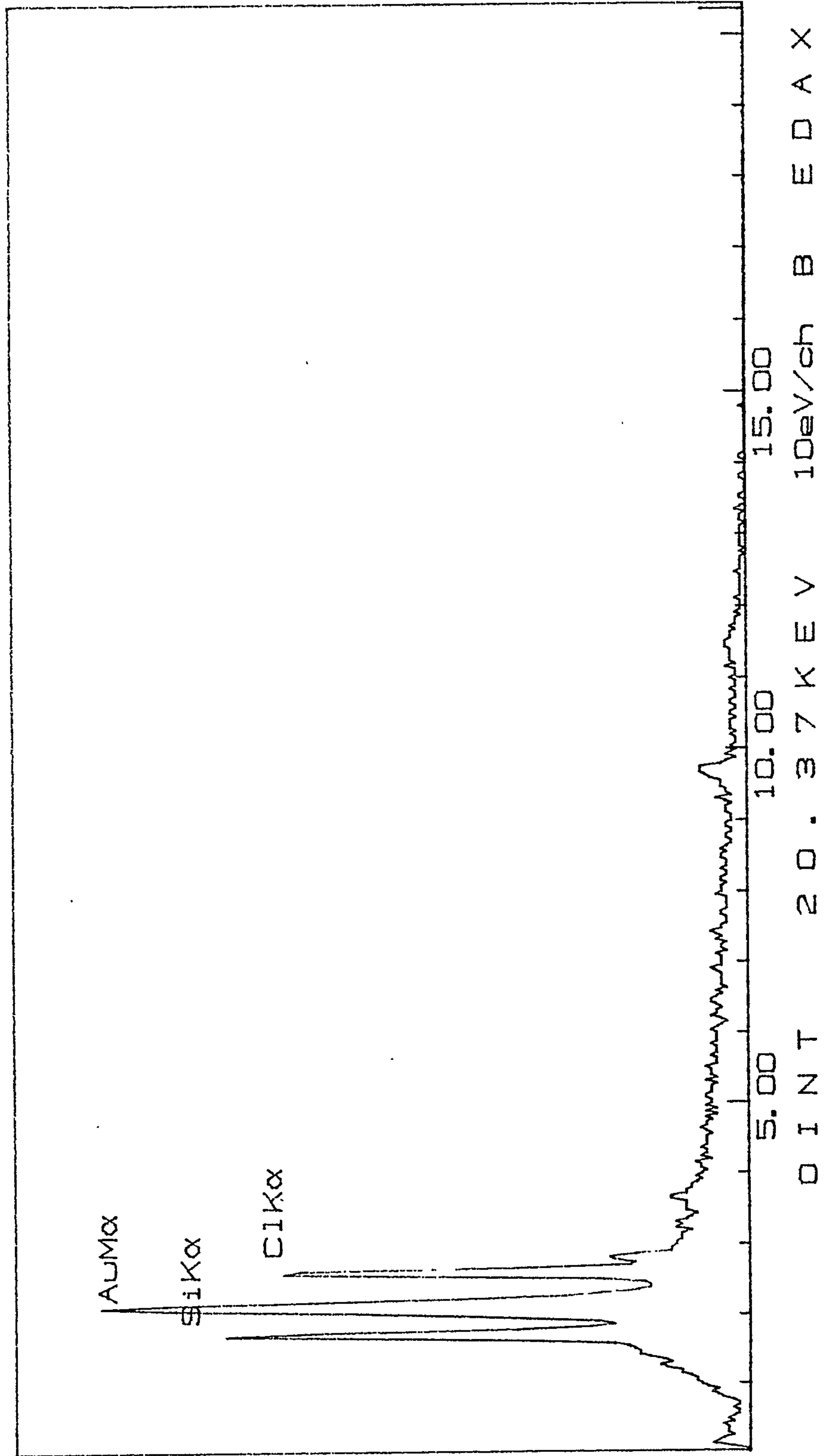


Figure 42 EDAX analysis of Fusion 24 hour specimen

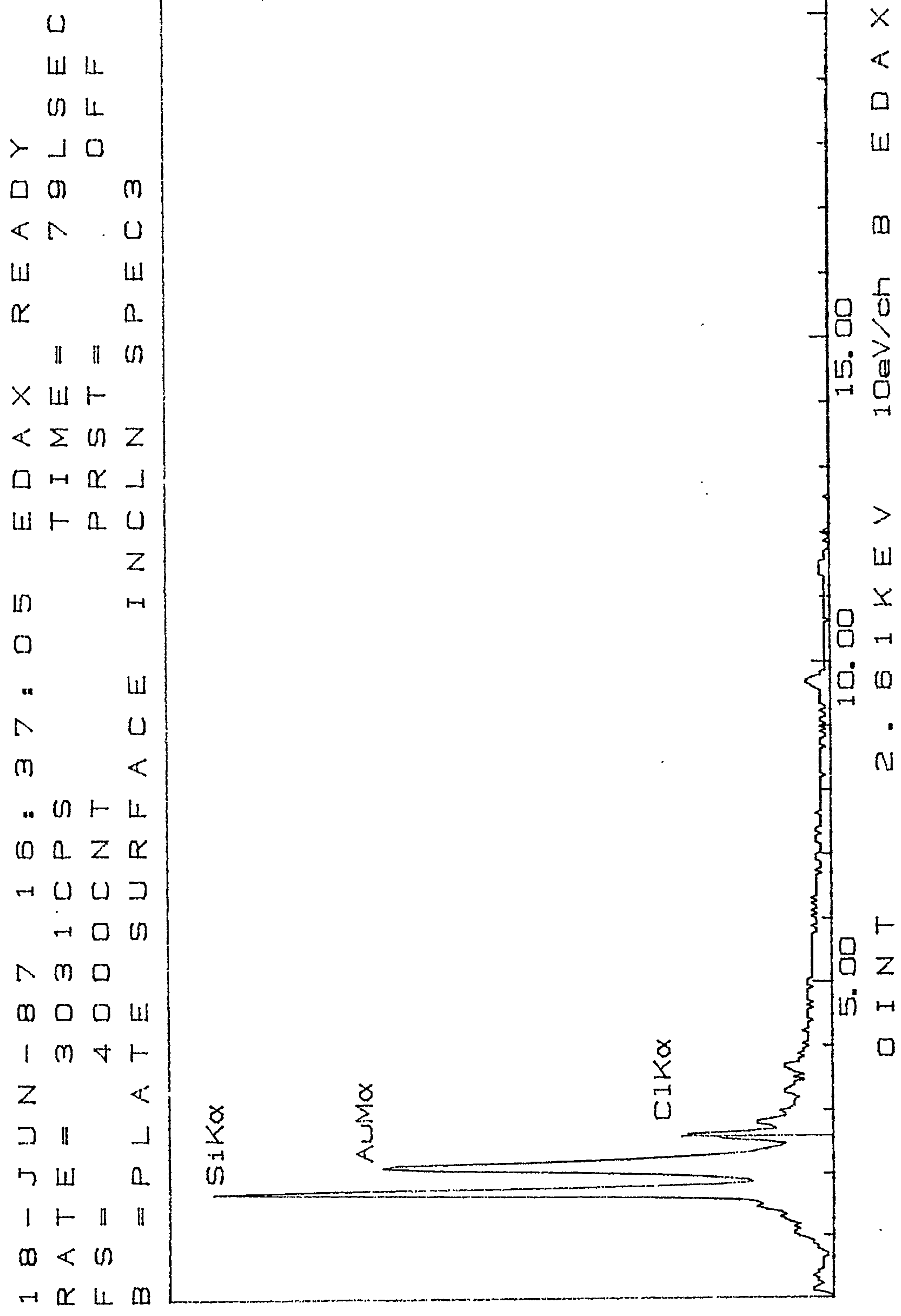


Figure 43 EDAX analysis of Panavia Ex 10 minute specimen

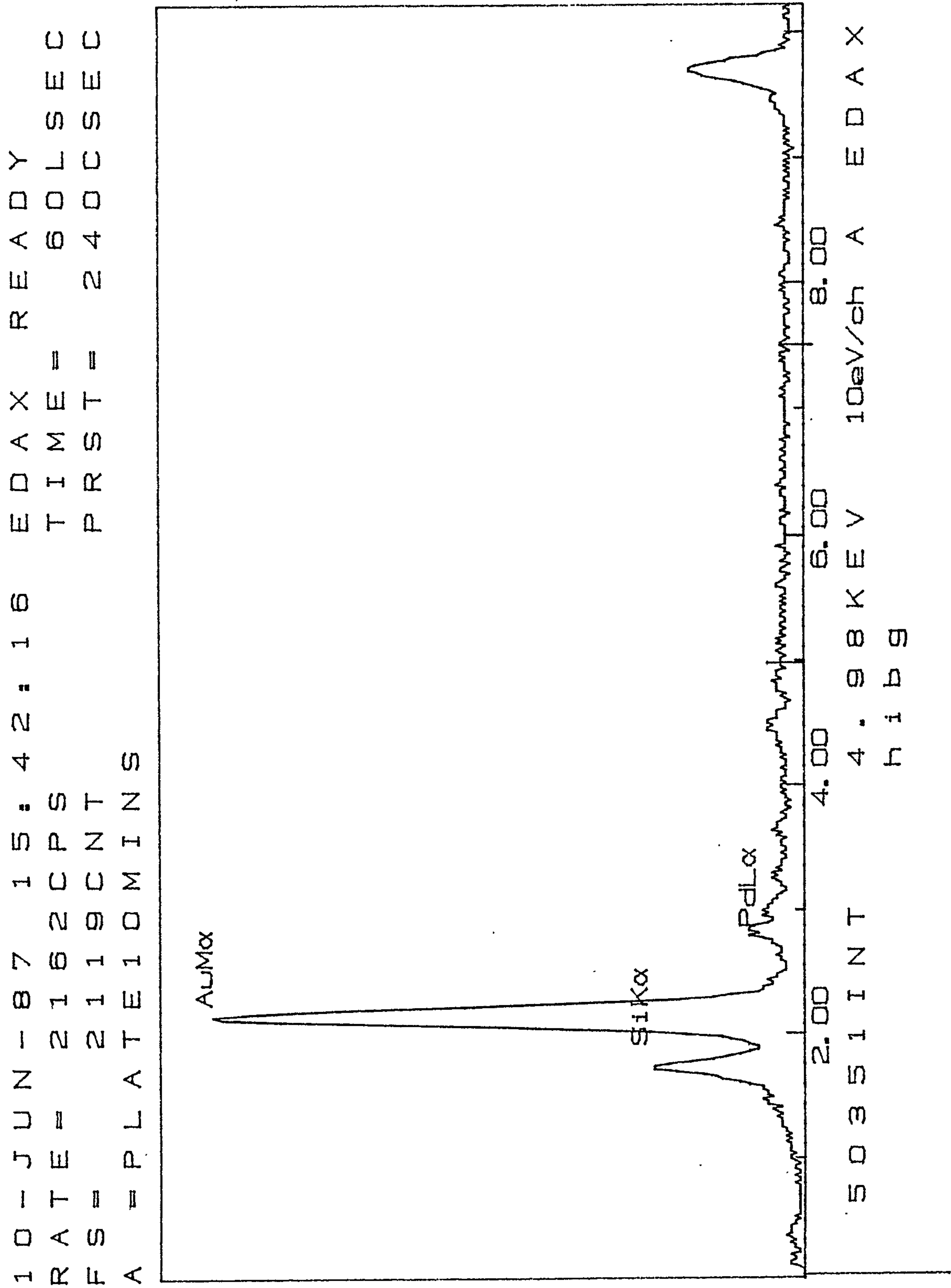


Figure 44 EDAX analysis of Panavia Ex 24 hour specimen

10-JUN-87 15:23:27 EDAX READY  
 RATE=376 CPS TIME=82L SEC  
 FS=2045 CNT PRST=240L SEC  
 A=PLATE INCL DARK3

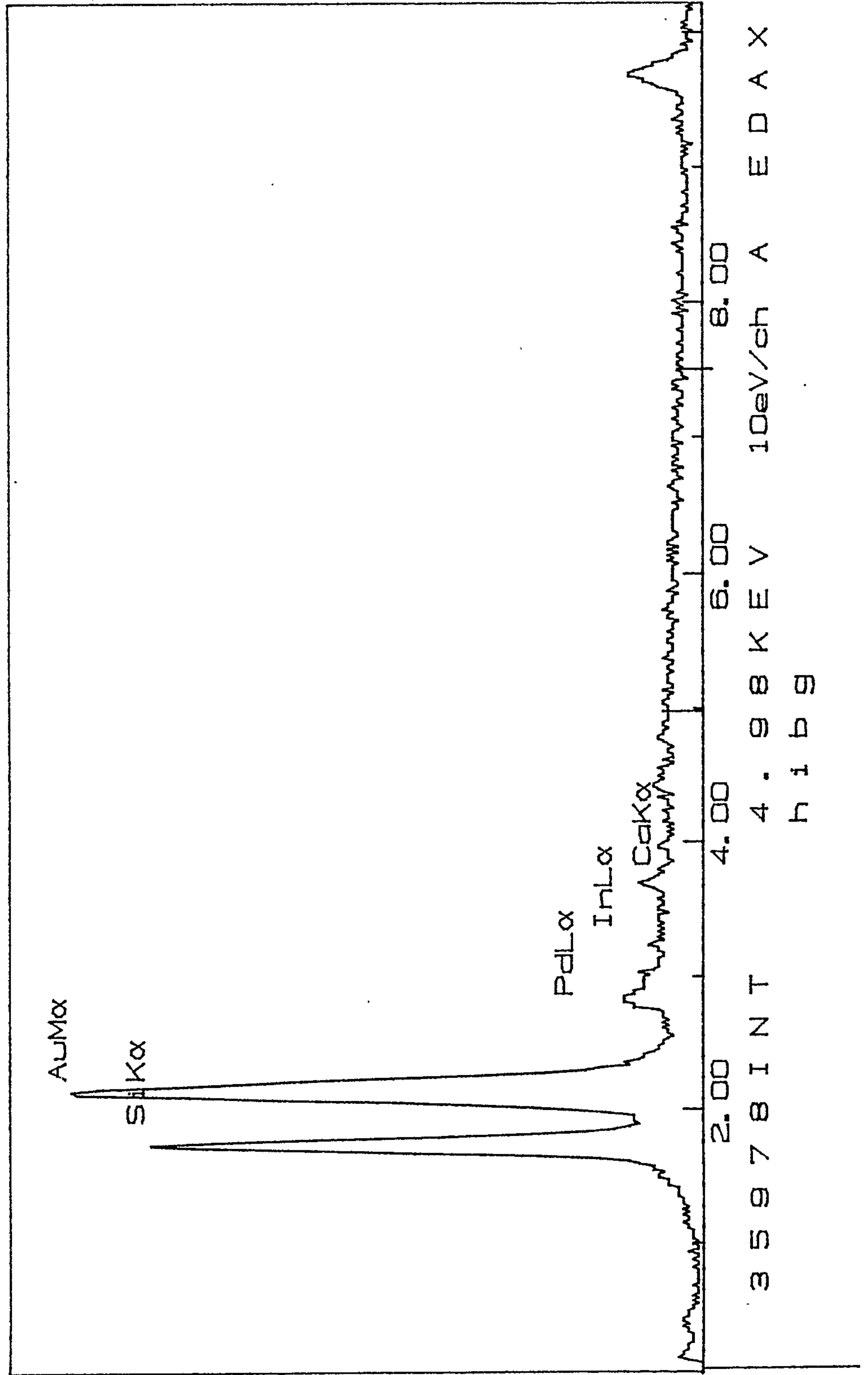


Figure 45 EDAX analysis of Scotchbond 10 minute specimen

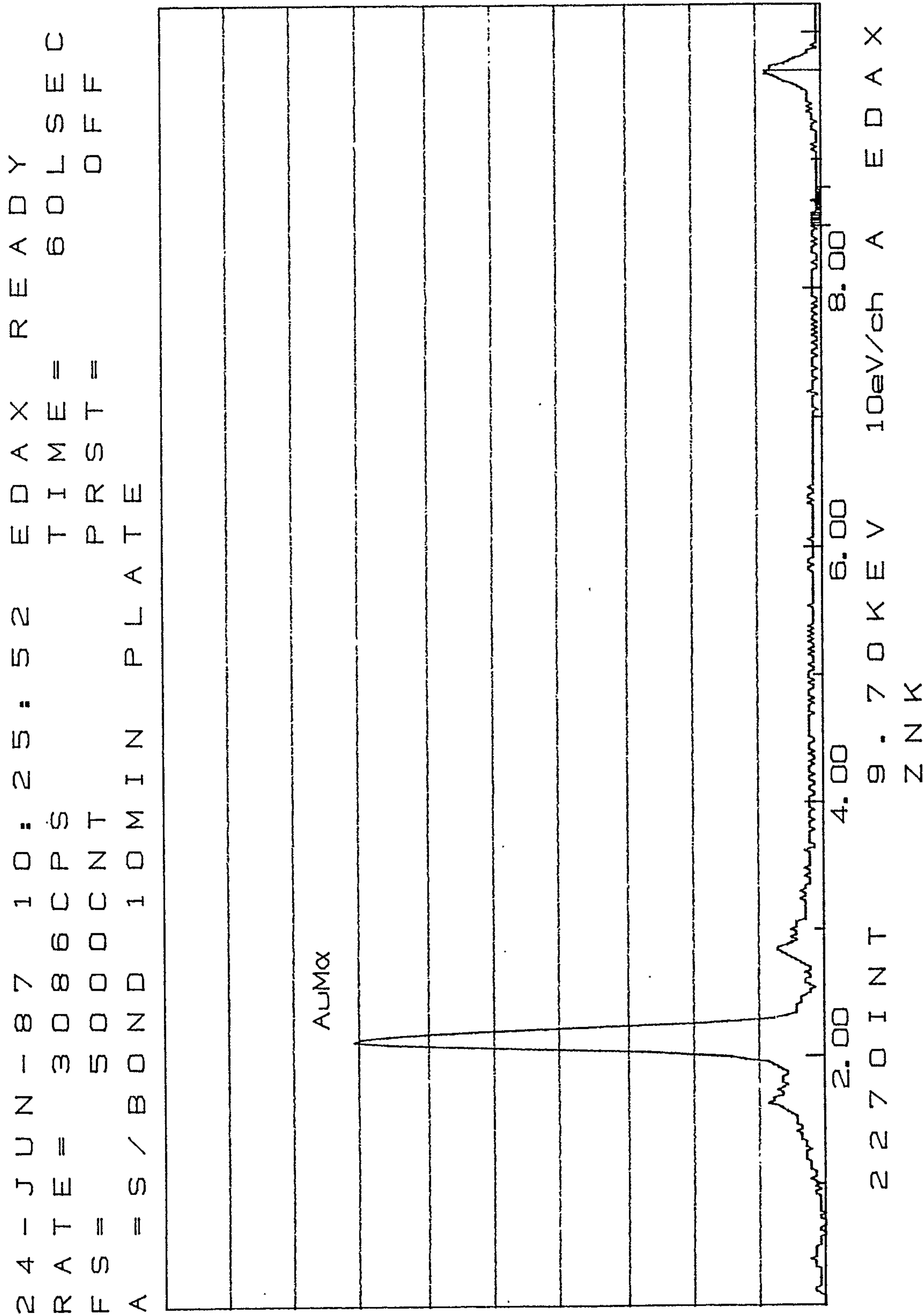


Figure 46 EDAX analysis of Scotchbond 24 hour specimen

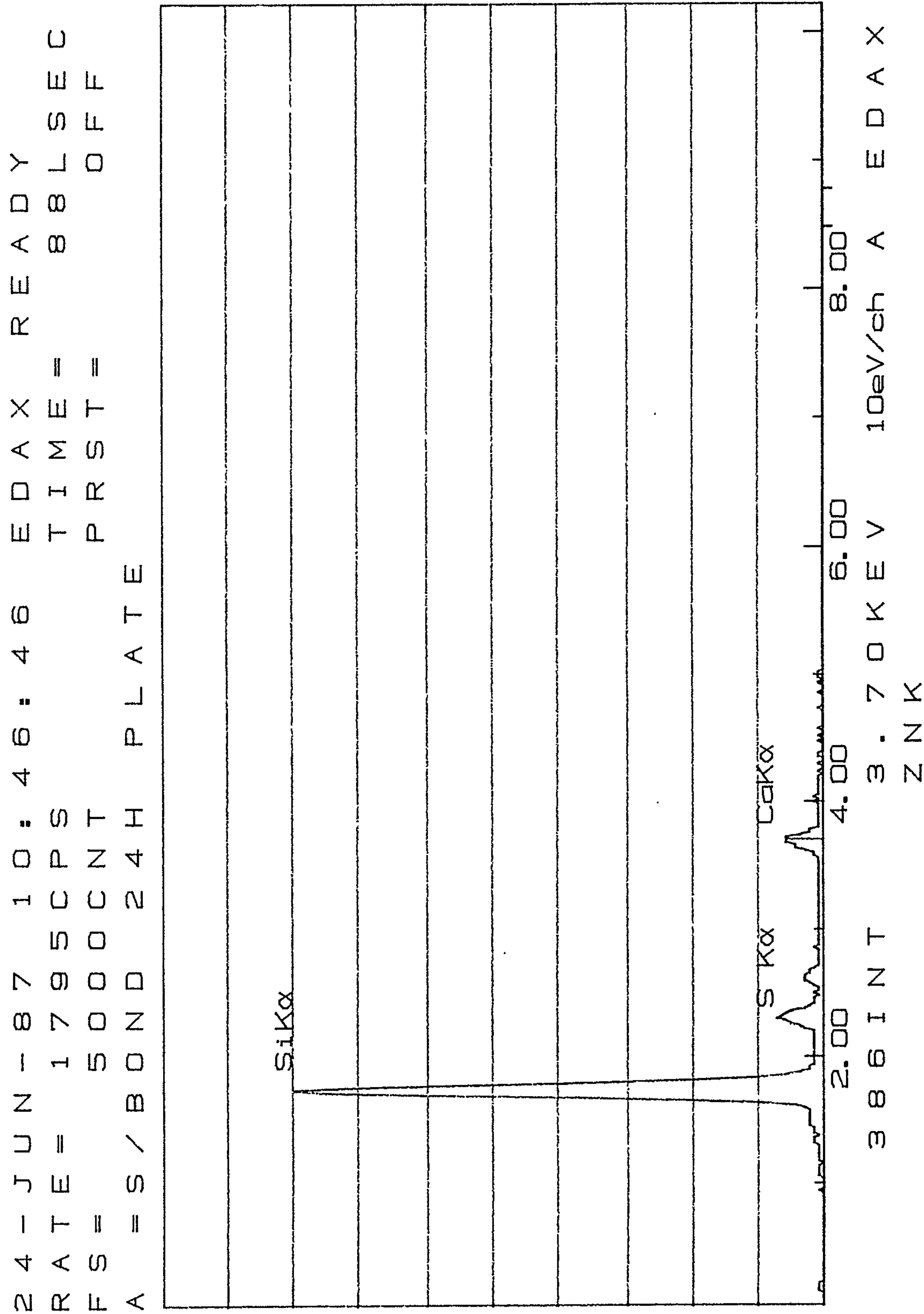


Figure 47 EDAX analysis of Scotchprime 10 minute specimen

15-SEP-87 12:33:06 EDAX READY  
 RATE= DCP S TIME= 44 LSEC  
 FS= 2284 CNT PRST= 200 CSEC  
 A=SP 10

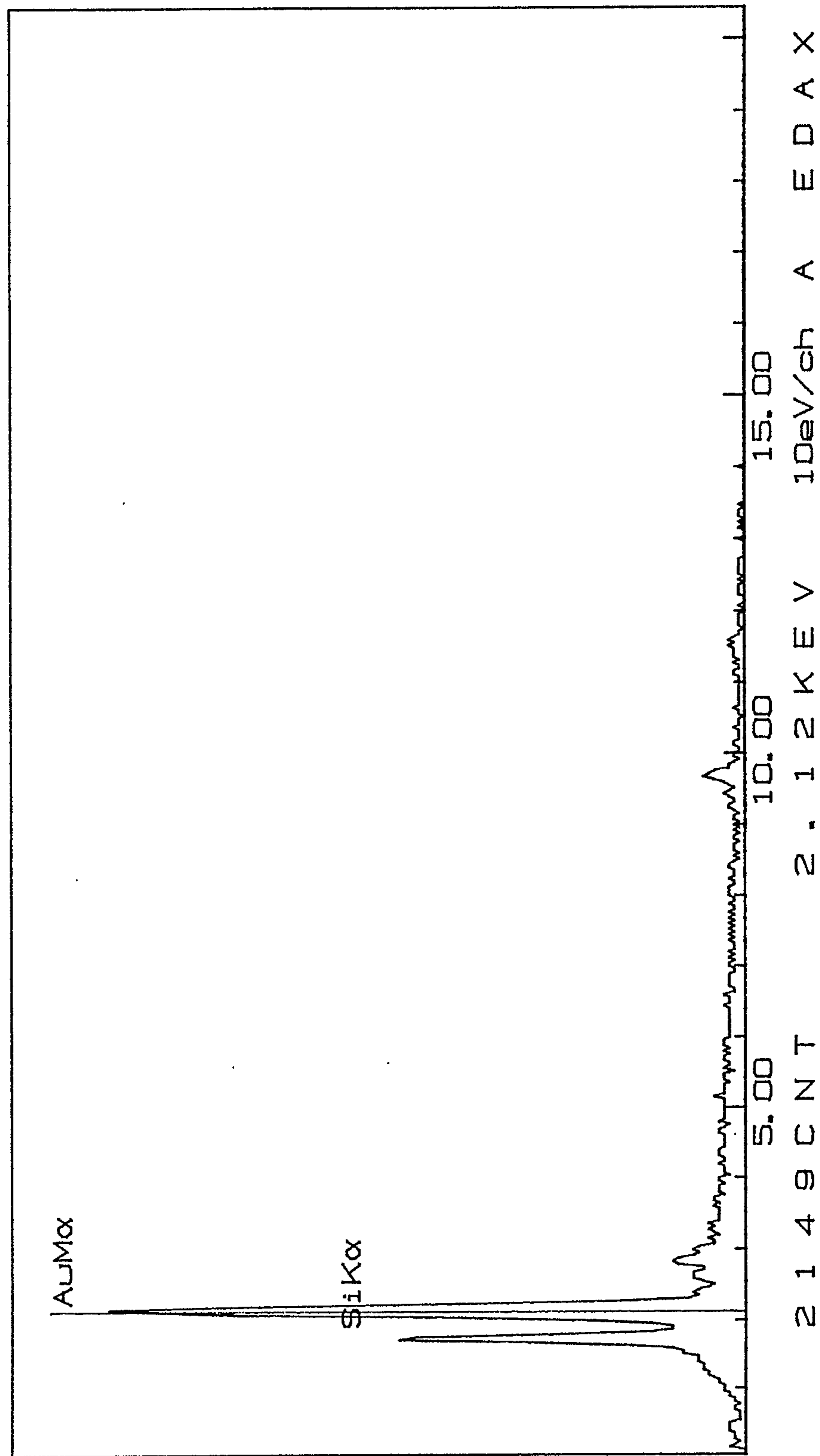


Figure 48 EDAX analysis of Scotchprime 24 hour specimen

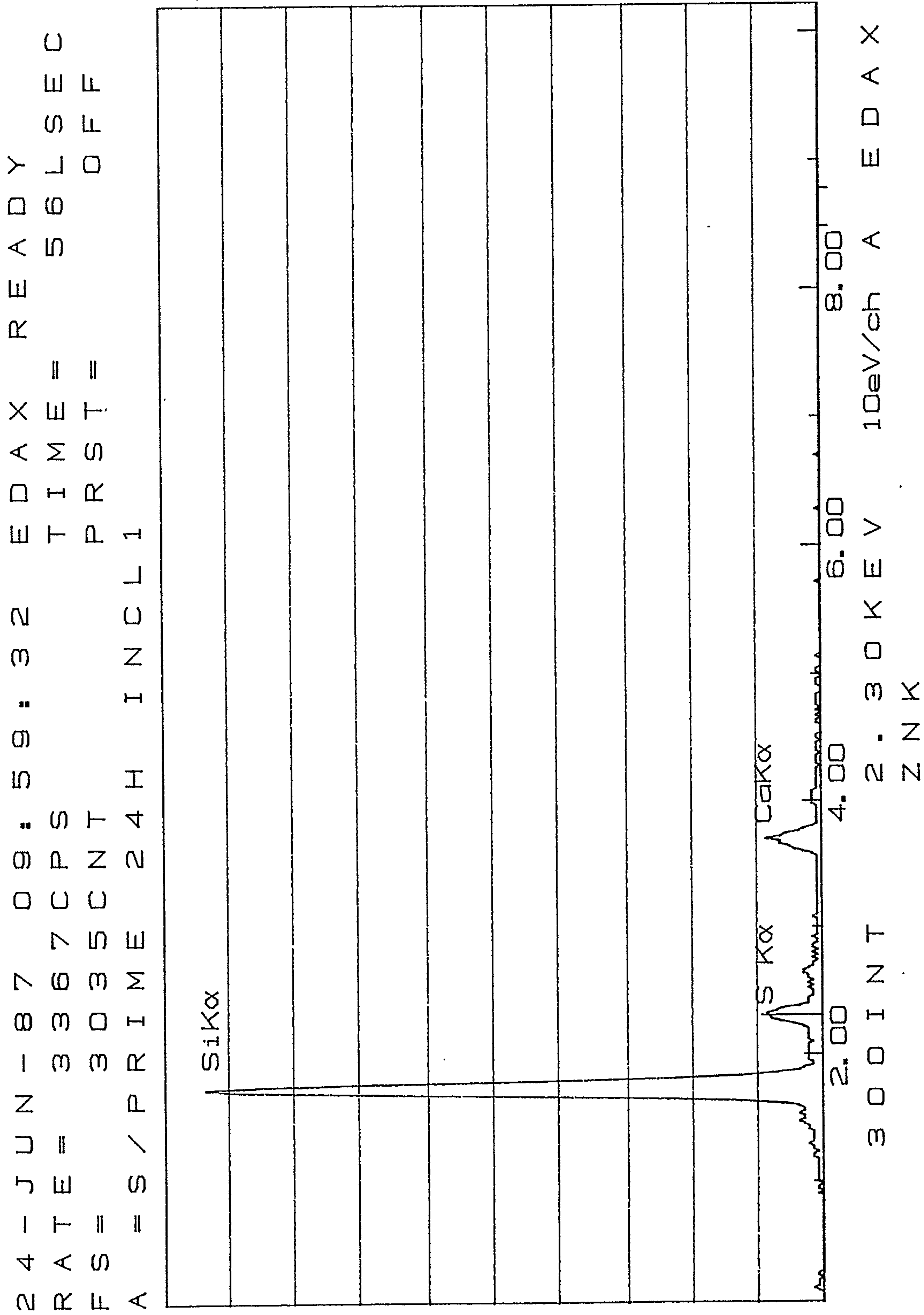


Figure 49 EDAX analysis of Scotchprime 24 hour lingual button

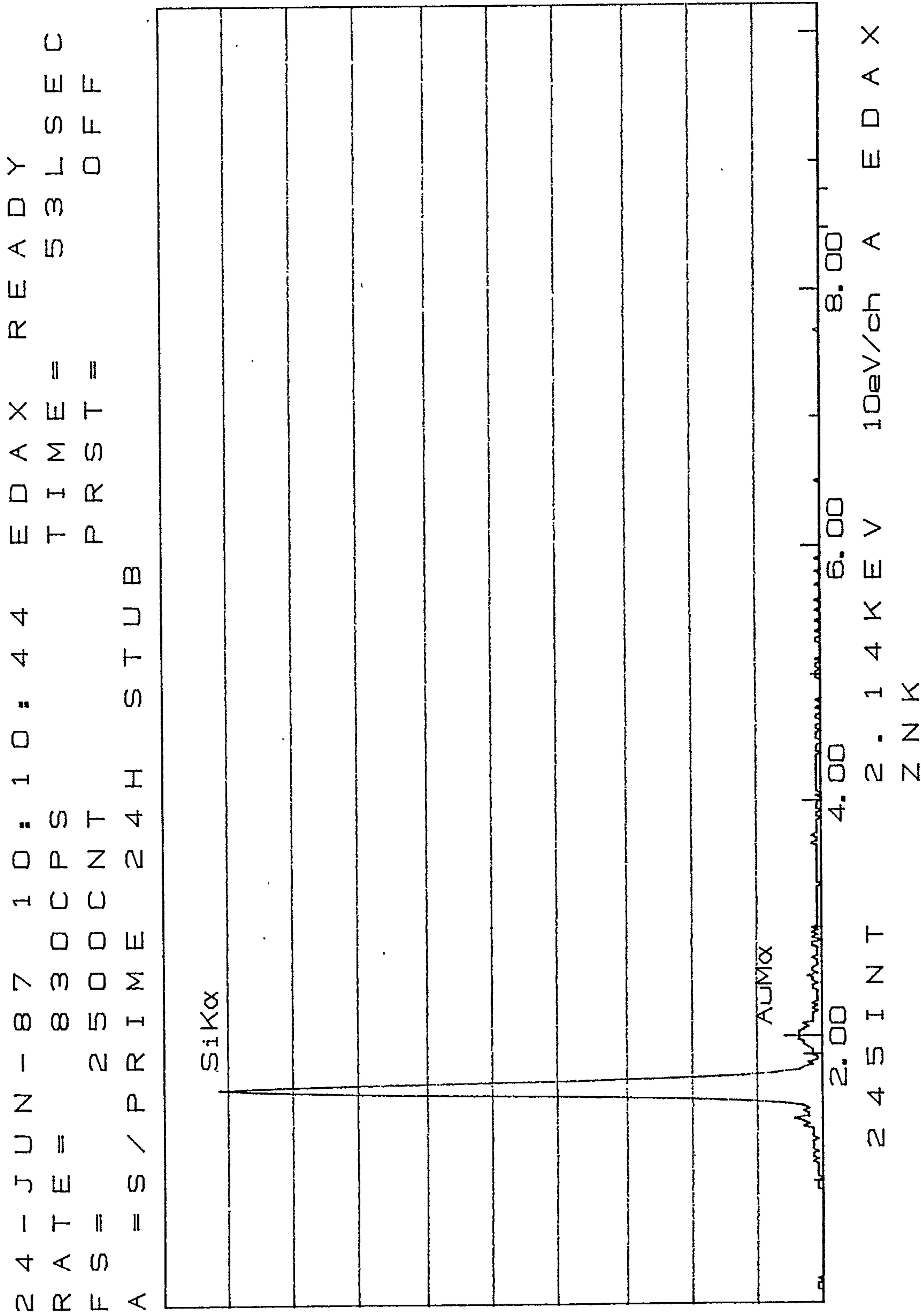
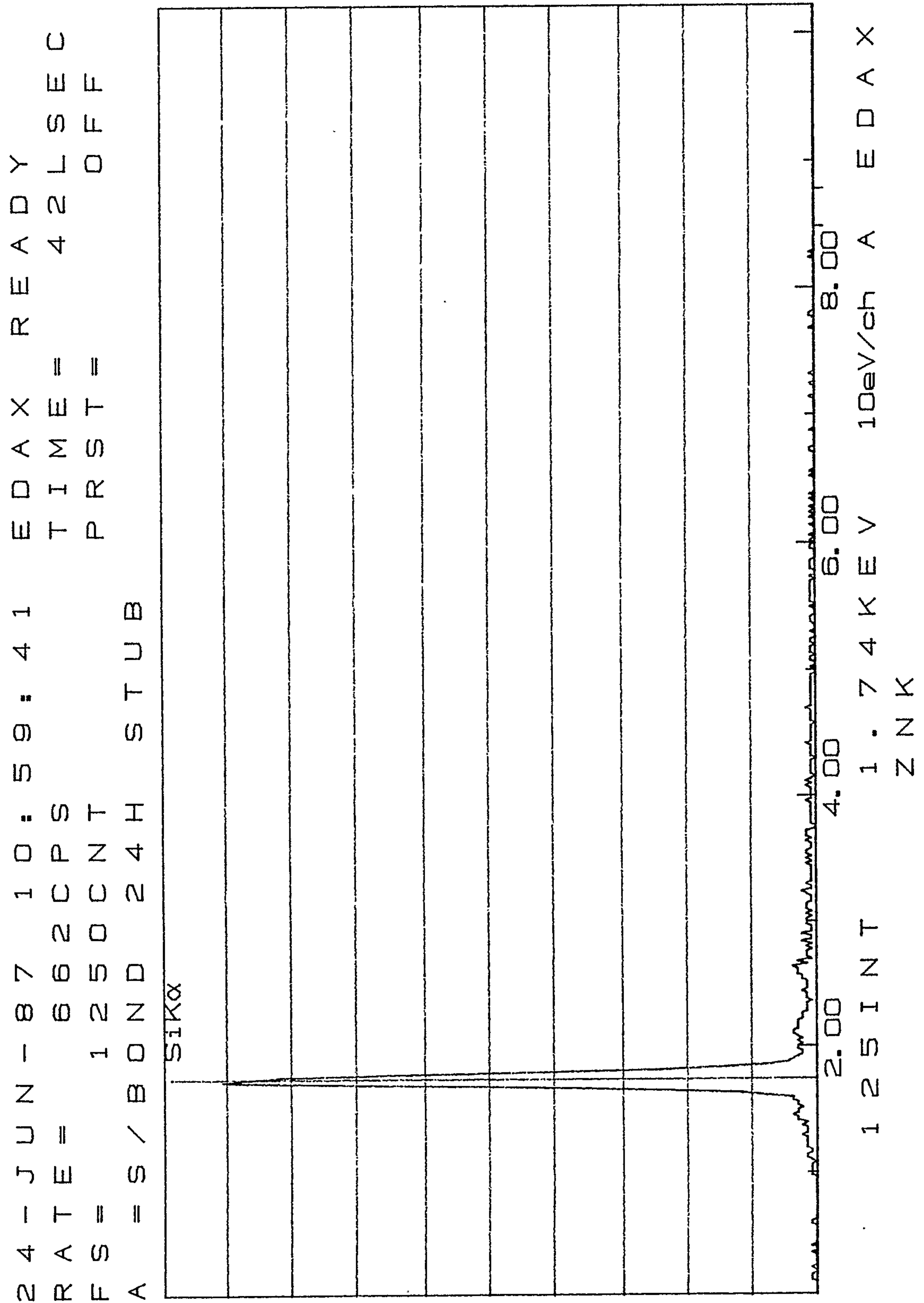


Figure 50 EDAX analysis of Scotchbond 24 hour lingual button



## 14. DISCUSSION

### **14.1. MATERIALS**

A number of investigators have reported laboratory results of orthodontic bonding systems to human enamel. The literature contains fewer reports of bonding to surfaces other than enamel. Ghassemi-Tary (1979) used Fusion to bond to porcelain and found the resulting bond test values to be within the acceptable range of forces commonly used in orthodontics.

Eames, Rogers, Feller and Price (1977) tested the effectiveness of Fusion as a gold bonding agent by bonding to uncast gold ingots. They found that Fusion did not improve the bonding of composites to gold and that the forces obtained in their bond strength tests were no higher than those achieved with unfilled resins only. They concluded that the manufacturer's claim that Fusion was capable of bonding to gold could not be substantiated.

The manufacturer of Scotchprime (3M), claims that the bond of this product to metal is dependent on the type of alloy used. In the bond tests cited in 3M's promotional literature they use the metal as supplied by the manufacturer and do not cast it. The bulk of 3M's claims are based on bond strengths to non-precious alloys and not to precious alloys recommended for crown and bridgework, such as Degudent U used in this study. The sample numbers in their product tests are also low at five, half the number of the present study. 3M claims that as a result of providing the silane molecule in its hydrolyzed form it enhances bonding to metal surfaces.

The nature of the silane bond to metal is somewhat uncertain. Noble metal alloys contain little, if any, surface oxides (Taub 1987). This could explain the manufacturers' claims for greater bond strengths to non-precious alloys which have an increased oxide content. The actual chemical bond to noble metal is probably very small.

#### **14.2. VARIATION**

In this study an attempt has been made to eliminate as many variables as possible. Unfortunately, it is not possible to eliminate all of the variables. The degree of variation found in the results, as exhibited by the co-efficients of variation, may be due to several factors (Keizer, ten Cate & Arends 1976), such as;

1) minor misalignment and deformation of the testing apparatus. Tweeddale (1965) states that pure shear stress is difficult to apply because bending and deformation of the shearing edge cannot be completely eliminated, in spite of careful machining and the use of high strength materials.

2) uneven adhesive film thickness.

3) voids within the adhesive. Jassem, Retief and Jamison (1981) point out that the introduction of air bubbles into a composite resin during mixing is an uncontrollable variable which may produce variation in bond strength results, as the air bubbles can act as areas of stress concentration.

4) inaccurate proportions of base and catalyst (Alexandre, Young, Sandrik & Bowman 1981).

5) age of bonding agent. Newburg and Pameijer (1978) reported, from their study of bonding composite resins to dental porcelain using a silane solution, that results produced from the use of an

old silane solution, were inconsistent and erratic. Thus the age and limited shelf life of the available silane solutions may introduce an additional variable.

Beech (1982) states that even with extreme caution during preparation and testing, co-efficients of variation of 30% or more are usually found. Brauer and Huget (1972) report co-efficients of variation of resin bond strength tests after 24-hours water exposure were of the order of 20 to 40%. Low, Davies and von Fraunhofer (1975b) report co-efficients of variation following bond tests after 7 days in water at 37°C, of 25%. Jassem, Retief and Jamison (1981) obtained co-efficients of variation ranging from 40 to 55% for shear strength tests after 24 hours of water exposure. In the light of these studies the present values of the co-efficients of variation appear relatively acceptable.

Unfortunately, the differences in experimental variables between operators means that comparison of values of bond strength between different workers is usually not meaningful. An exception to this may be a comparison of the present study to that done by Bassier (1984) which was conducted in the same laboratory using the same shear testing apparatus and the same time scale, for an enamel-bonded, acid-etch technique. Bassier obtained values at debonding of 10 MPa for 10 minutes and 20 Mpa for 24 hours, to human tooth enamel.

All manufacturers' recommend roughening of the gold surface prior to bonding to enhance bond strength. This introduces an additional variable into the experiment. Jochen and Caputo (1977) found that increasing the degree of surface roughness of an adherend leads to increases in bond strength.

### 14.3. FAILURE SITES

Faust, Grego, Fan and Powers (1978) and Maijer and Smith (1981) noted from their studies that most bonded systems using metal brackets failed at the bracket-cement interface. Jassem, Retief and Jamison (1981) also found failure predominantly at the bracket-resin interface but stated that failure at this site is not a true reflection of resin bond strength.

In contrast, Zachrisson (1977), Lopez (1980), and Knoll, Gwinnett and Wolff (1986) note that the failure sites in their studies occur predominantly at the resin/substrate interface. This is also the case in the present study. Thus it would appear that the values obtained are an accurate reflection of the true bond strength and not merely an indication of the cohesive strength of the adhesive.

Retief (1974) points out that sites of failure cannot be reliably classified by visual or low magnification microscopic examination. The only accurate method of bond site failure detection is by the use of a SEM. However, the remnants of adhesive observed under the SEM in this study may not be evidence alone of a true adhesive bond. Causton (1982), advises that isolated areas of adherent cement do not indicate cohesive failure, but simply that the fracture crack resulting from bond testing may have reached a very high speed and begun to bifurcate.

### 14.4. TEST VALUES

As would be expected, the shear strength values in this study were greater at 24 hours than the values at 10 minutes, despite the period of water immersion. Crabb & Wilson (1971)

note that this is because the setting reaction has gone further towards completion. Craig (1981) explains that the polymerization reaction of composite continues long after the setting time is reached and has been known to continue up to 24 hours after initial hardening.

Retief, Dreyer and Gavron (1970) maintain that under clinical conditions, it is possible to keep the bonding resin on the teeth relatively dry for a period of up to 10 minutes. For this reason the 10 minute test specimens in this study were not subjected to water immersion.

#### **1.4.5. BOND STRENGTH**

Smith and Maijer (1983) feel that bond strength under shear stress should be well above the minimum required to resist failure, but do not specify to what extent. The mean bond strength required of an adhesive is difficult to determine, since occlusal loads vary considerably and an assessment of the force transmitted to an individual tooth is difficult to obtain (Lopez 1980). Previously it had been stated that the average bond strength minus three times the standard deviation should be equal to the maximum force exerted on a bracket during the orthodontic treatment (Keizer, ten Cate & Arends 1976). Guzman, Faust and Powers (1980) state that cements which form a stronger bond in vitro presumably also form a stronger bond in vivo.

The nature of adhesion to gold surfaces is still unclear. Barquins and Cognard (1986) examining the adhesion of epoxy resin to gold found that it occurred essentially by the vehicle of a low energy dispersive interaction and hence concluded that it must take place through the medium of a low energy superficial

layer on the metal. This could explain the poor nature of the bond to what should otherwise be a receptive high energy surface.

#### **14.6. USE OF A SILANE PRIMER**

Faulkner and Harcourt (1975) found that the strength of the bond between an acrylic resin and stainless steel treated with a silane coupling agent was stronger than when the silane coupling agent was not used. However, Beech and Jalaly (1980) concluded that a silane primer made no significant difference to the bond strength of an orthodontic bonding material in their study.

From the results of the present study this author must agree with the conclusion of Beech and Jalaly (1980). It appears the use of a silane priming agent (such as Scotchprime or Fusion), or a metal bonding composite (such as Panavia Ex), to enhance an orthodontic bond to precious dental gold alloy does not significantly improve this bond when compared with a conventional composite bonding material.

## **15. CONCLUSION**

This study demonstrates that:

- 1) It is possible to orthodontically bond to a high gold content crown and bridgework dental alloy. However, the shear bond strength values are only approximately one half of comparable bond strengths using an acid-etch technique to human enamel.
- 2) The 24 hour shear bond strength values of all the materials examined are within clinically acceptable limits according to the dominant literature regarding maximum orthodontic force levels. Bond strength however, may not be sufficient to withstand masticatory forces generated on lingual orthodontic appliances.
- 3) The weakest point in the gold bonded joint was at the adhesive/substrate interface.
- 4) The greatest initial bond strength is obtained by using Panavia Ex. After a 24 hour period, however, there is no difference in bond strength between any of the materials tested.
- 5) Scotchprime at 24 hours, as examined by scanning electron microscopy and light microscopy, exhibited an increased number of adhesive inclusions when compared to all other specimens. Traces of gold attached to the bonding resin of the corresponding lingual button of Scotchprime only, promote the suspicion that Scotchprime may be the most effective gold bonding agent of those tested. Unfortunately, this increased bonding ability did not translate into increased bond strength. The number of sites

available on the gold surface for chemical bonding may be insufficient to allow formation of a substantial chemical bond. A larger sample would need to be examined under SEM to verify this suspicion.

6) If there is no difference between materials in bond strength at 24 hours, it may be more cost-effective to employ a conventional bonding agent, such as Scotchbond, rather than one of the specialised gold bonding agents.

7) It is not possible to determine, from visual examination alone, the site of bond failure of an adhesive.

A recently developed adhesive, 4-methacryloxyethyl trimellitate anhydride, (4-META)/MMA-TBB resin, may be more effective as a gold bonding agent than the materials tested in this study (Mogi 1982; Stokes 1987). Further research is required in this area to produce a more effective gold bonding agent.

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APPENDIX A**GLOSSARY OF TERMS USED IN CHAPTER 4**

|                   |                                                                                                                                                  |
|-------------------|--------------------------------------------------------------------------------------------------------------------------------------------------|
| Alcohol           | Compound of the general formula R-OH.<br>Denoted by the suffix -ol.                                                                              |
| Alkyl             | Hydrocarbon groups of the general formula $C_nH_{2n+1}$ containing one less hydrogen than the parent group.                                      |
| Alkoxy            | Hydrocarbon groups of the general formula $OC_nH_{2n+1}$                                                                                         |
| Anhydrous         | The absence of water.                                                                                                                            |
| Condensation      | This is a reaction in which the elements of water are removed from functional groups.                                                            |
| Ester             | Compound produced by the reaction between an alcohol and an acid which has the general formula; $\begin{array}{c} R-C-OR \\    \\ O \end{array}$ |
| Esterification    | The reaction between an alcohol and an acid<br>$RCOOH + R'OH \rightleftharpoons RCOOR' + H_2O$<br>This is a reversible reaction.                 |
| Ether             | Compound of the general formula R-O-R, in which an oxygen bridge joins two hydrocarbon groups.                                                   |
| Hydrolysis        | Any reaction with water.                                                                                                                         |
| Hydroxy group     | An -OH group which characterizes both alcohols and phenols.                                                                                      |
| Monomer           | The simplest molecular form of a substance.                                                                                                      |
| Organic           | Compound containing carbon.                                                                                                                      |
| Organo-functional | Portion of organic compound which undergoes change during a chemical reaction.                                                                   |

|               |                                                                                         |
|---------------|-----------------------------------------------------------------------------------------|
| Oligomer      | Short chain polymer.                                                                    |
| Oxane bond    | A bond formed by oxygen in the place of carbon<br>i.e. $R-O-R$ .                        |
| Polymer       | The compound formed by the joining together<br>of numerous simple molecules (monomers). |
| Silanetriol   | Silane molecule with three hydroxy<br>functional groups.                                |
| Thermoplastic | These materials soften when heated.                                                     |
| Thermoset     | These materials do not melt.                                                            |

APPENDIX B**BOND STRENGTH CONVERSIONS**

Bond strengths have previously been expressed in lb/in<sup>2</sup> or kg/cm<sup>2</sup>. During shear stress tests force and not mass is being measured and it is therefore more correct to express the bond strength in N/mm<sup>2</sup> or MPa (Retief 1974).

The conversions can be performed as explained below;

$$1 \text{ Newton} = 1 \text{ pound} \times 4.448$$

$$1 \text{ Newton} = 1 \text{ kg force} \times 9.807$$

$$1 \text{ kg/cm}^2 = 0.0981 \text{ N/mm}^2$$

$$1 \text{ N/mm}^2 = 1 \text{ MPa}$$

APPENDIX C

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