THE SPRING RESEARCH AND MANUFACTURERS' ASSOCIATION

PROBLEMS ASSOCIATED WITH ELECTROPLATED SPRINGS

Report No. 405

by

L. F. Reynolds

DECEMBER 1986

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SUMMARY

Unsatisfactory electroplated surface finish is an occasional, but too-frequent problem for springmakers. Following a survey of these problems a number of case histories were studied in detail. Optical and scanning electron metallographic techniques revealed the causes of the surface finish problems, such as bubbles, black marks, dull appearance and stains. The majority of faults were attributable to inadequate electroplating process controls, and relatively few faults were due to material faults or inadequate cleaning prior to electroplating.

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1. INTRODUCTION

A significant proportion of carbon steel springs are electroplated to confer corrosion protection and an attractive appearance, or other special properties such as good solderability.

Over a period of several years, many SRAMA members have experienced rejections of electroplated springs due to poor quality of the plated deposit. The problems appear to be most severe where plating is sub-contracted out, although members with "in house" electroplating facilities also experience some problems.

This report presents the results obtained at SRAMA during a detailed examination of electroplated springs following a survey of members' electroplating problems. The survey indicated that rejections of electroplated springs occurred for five main reasons:

i. Blistered plate, often with poor adhesion

ii. Pitted plate

iii. Rough plate

iv. Dull plate

v. Discoloured plate, or plate of non-uniform appearance.

The plated components were submitted by SRAMA springmaking members as being typical of the problems they encountered.

The samples consisted of strip springs made from 0.45-0.75mm thick material, compression springs made from 0.7-3mm diameter wire and one wire form made from 4.7mm diameter mild steel wire.

The report is not an exhaustive treatise of electroplating practice, which is fully documented in the literature, but rather is a practical record of some typical plating problems and the probable causes of and cures for these difficulties. A brief bibliography of appropriate publications is given at the end of the report.

2. SAMPLES RECEIVED AND EXAMINATION TECHNIQUES

The identification of the plated steel samples is shown in Table I. The SRAMA project identification has been retained for future identification.

Two main techniques were employed, namely optical and scanning electron microscopy. The scanning electron microscope was used for the greater part of the work, since this technique was much more suited to detailed examination of small areas on the plated surface. The great depth of focus provided by the SEM was particularly valuable during this work.

The results are presented in the form of individual case histories, and are organised with respect to plating, as shown in Table I.

3. CASE HISTORIES

3.1 Blistering of nickel plate

3.1.1 Plating Problem

The austempered steel clips had been bright nickel plated. There was considerable blistering of the nickel plate. The nickel deposit was broken away from the clip at some blistered zones, to reveal the underlying steel surface.

3.1.2 Detailed Examination

The steel surface beneath the blisters was examined on the scanning electron microscope. Numerous pits were present in the steel surface at these locations, as shown in Figs 1 and 2. Subsequent enquiries revealed that, prior to plating, one of the pre-cleaning operations involved a room temperature pickle in uninhibited 50% hydrochloric acid.

3.1.3 Conclusion

The steel surface was severely pitted by a 50% hydrochloric acid pickle, leading to poor adhesion and blistering of the nickel during electroplating.

3.1.4 Recommendation

Springs should not be pickled in 50% hydrochloric acid. Preliminary surface preparation can be accomplished using alkaline or anodic cleaning techniques, as specified in DEF 03-2 for example. Subsequent acid cleaning should be carried out in hydrochloric acid solutions not exceeding 20% concentration, and suitable inhibitors should be used to minimise chemical attack of the steel surface during acid cleaning.

3.2 Dull/pitted nickel plate

3.2.1 Plating Problem

The conical springs were bright nickel plated. Optical examination of the springs showed two clear categories of defective plating.

i. Dull and brown/purple discoloured areas.

ii. Small localized unplated areas, often associated with a red/brown greasy deposit within the unplated area.

3.2.2 Detailed Examination

i. Dull/discoloured areas

These two areas of plate displayed essentially similar characteristics, as shown in Figs 3-5.

The dull areas had a coarse granular appearance (Figs 3 and 4).

The discoloured areas also exhibited a granular appearance, but the granularity was much finer than that of the dull plated areas (Fig 5).

ii. Localised unplated areas

The greasy deposit was chemically removed to prevent damage to the scanning electron microscope.

A typical unplated zone is shown in Fig 6. The unplated wire surface was clearly visible at the bottom of a wide, shallow pit in the nickel plate.

3.2.3 Conclusion

The defects suggest that the plating baths were contaminated with suspended material.

The wide, shallow pits may have resulted from organic contamination of the bath, or from incomplete solution of an organic leveller such as Coumarin during the plating process.

3.2.4 Recommendation

The problems can be corrected by correct pre-cleaning, bath maintenance and filtration procedures for bright nickel plating.

3.3 "Spotting" of nickel plate

3.3.1 Plating problem

The austempered spring clips had been bright nickel plated. The flat strip components were slightly bowed in shape, resulting in the formation of concave and convex surfaces. The convex surface was satisfactorily plated, but brown "spotting" was evident on much of the concave surface.

3.3.2 Detailed Examination

Scanning electron microscopy revealed numerous pits in the nickel plate on the concave spring surface, as shown in Fig. 7 Many of the pits were very small, being less than 0.0005mm in diameter.

Optical measurement of the nickel plate thickness gave the following results:

Convex surface	Concave surface
0.01 - 0.015mm	0 - 0.008 mm

The spring steel material was satisfactory, with no evidence of surface defects or non-metallic inclusions at the strip surface.

3.3.3 Conclusion

Relatively poor throwing power during bright nickel plating gave a slower rate of nickel deposition at the concave surface. The resulting plate at this location was too thin to completely cover the steel surface, leaving numerous small pits in the nickel deposit. The plating solution in the pits was not completely removed during washing, resulting in the development of spotting stains on the plated springs.

3.3.4 Recommendation

The springs should be nickel plated to give a minimum thickness of at least 0.01 mm nickel at the concave surface.

3.4 Pitted chromium plate

3.4.1 Plating problem

The components were made from mild steel wire and had been chromium plated after a copper flash. The wire forms showed evidence of severe pitting on most of the chromium plated surface.

3.4.2 Detailed examination

Scanning electron microscopy revealed that the pits extended completely through a very thick chromium deposit, as shown in Figs 8 and 9. More detailed examination indicated that the pits in the chromium plate initiated at small intrusions in the steel surface, as shown in Fig 10. These findings were verified by optical microscopy, which confirmed that gas pitting of the chromium plate was initiated by 0.035mm deep intrusions in the steel wire surface, as shown in Fig 11. Optical microscopy further showed that the chromium plate was 0.09mm thick, wich is substantially greater than the 0.0002-0.002mm deposit normally used for bright chromium plate.

3.4.3 Conclusion

Hydrogen pitting of the chromium plate was initiated at intrusions in the drawn mild steel surface.

The copper flash did not give any significant levelling action to provide the smooth, bright surface necessary for bright chromium plating.

Gas pitting was further accentuated by a chromium layer which was approximately 0.09mm thick.

3.4.4 Recommendation

Similar gas pitting can be avoided by copper flashing, and then bright nickel plating the parts in a bath with good levelling action to give a minimum nickel thickness of 0.01mm.

A final chromium coating of 0.0002-0.002mm will provide the bright finish required for decorative applications.

3.5 Dull chromium plate

3.5.1 Plating problem

The spring was made from hard drawn spring steel wire. Of the 52 coils in the spring, 42 were close coiled whilst the remaining 10 coils at one end of the spring were of open pitch.

The spring had been given a flash coating of nickel followed by a bright chromium finish.

The close coiled sections of the spring were satisfactorily plated with bright chrome, but the open coiled section showed areas of dull chrome over 50-75% of the spring circumference.

3.5.2 Detailed examination

Examination of the close coiled area of the spring on the scanning electron microscope revealed a satisfactory deposit of bright chromium, as shown in Fig 12. There was little evidence of micro-cracking in the bright chromium, suggesting that the spring may have been chromium plated at a rather higher than normal current density.

Examination of the open coiled area of dull chrome revealed the granular chromium normally associated with a burned deposit, as shown in Fig 13.

The bright chrome area merged gradually with the burned area, as indicated by the appearance of an intermediate zone shown in Fig 14.

3.5.3 Conclusion

The dull chromium most probably resulted from a locally high current density at the open coiled zone of the spring.

3.5.4 Recommendations

A burned chromium deposit can be avoided by careful control of the voltage applied during chromium plating. In particular, components exposing a variable surface area to the plating solution will require careful control of the chromium plating process.

3.6 Rough zinc plate

The zinc deposit was very rough and granular over the whole surface of the compression spring.

3.6.2 Detailed examination

Examination on the scanning electron microscope revealed a very granular zinc coat of uneven distribution, as shown in Figs 15 and 16. Optical microscopy confirmed that the zinc was very unevenly distributed, with a deposit thickness varying between 0-0.15mm. Approximately 50% of the spring surface was not coated with zinc at all.

3.6.3 Conclusion

The granular appearance and poor distribution of the zinc suggests that the spring was plated in a bath low in metal/cyanide/hydroxide. The plating current may also have been higher than that normally employed for bright zinc electroplating.

3.6.4 Recommendation

Improved process control during plating will ensure that the bath composition, temperature and plating current remain in balance for bright zinc electroplating.

3.7 Dull zinc plate

3.7.1 Plating problem

The zinc plated compression spring was generally dull and lustreless. However, there was no visible evidence of granular zinc at any region of the spring surface.

3.7.2 Detailed examination

Examination on the scanning electron microscope revealed a lightly roughened zinc surface with some evidence of nodular zinc growth, as shown in Figs 17 and 18. Optical examination revealed an evenly distributed zinc deposit with a thickness of 0.02mm.

3.7.3 Conclusion

The combination of fine scale roughness and nodular zinc growth suggests that the plating solution was both out of balance and contaminated with suspended solids, possibly from anode sludge, dust etc.

3.7.4 Recommendation

Improved process control and filtration during plating will give the required bright zinc finish.

3.8 Blistered zinc plate

3.8.1 Plating problem

The zinc deposit was dull and blistered generally over the compression spring surface. Some of the blisters were severely cracked in several areas.

3.8.2 Detailed examination

Examination on the scanning electron microscope revealed a severely cracked and blistered zinc plate, as shown in Fig. 19.

More detailed examination showed the zinc to exhibit a marked nodular character, as shown in Fig 20.

Optical examination indicated that the spring was plated with two deposits. The first zinc deposit was 0.025mm thick with an even distribution, and showed good adhesion at the steel/zinc interface.

The second (top) zinc deposit was 0.04mm thick, and was unevenly distributed showing very poor adhesion at the zinc/zinc interface of the two deposits.

3.8.3 Conclusion

The nodular/blistered character and duplex nature of the zinc plate suggests that an unbalanced bath composition and poor process control was the cause of this plating problem.

3.8.4 Recommendations

Improved process control and attention to the bath composition during plating should eliminate the problem.

3.9 Blistered cadmium plate

3.9.1 Plating problem

The austempered steel strip sample showed marked blistering of the yellow passivated cadmium plate. The blisters occurred over a 4mm diameter zone at one end of the spring only, and were present on both sides of the spring at this zone.

However, the remaining 90% of the cadmium plate on the sample was completely satisfactory.

3.9.2 Detailed examination

The sample was examined on the scanning electron microscope and the general appearance of the bubbled zone is shown in Fig 21.

Detailed examination of the bubbled areas showed clear evidence that the cadmium plate was broken up by hydrogen gassing, as shown in Fig 22. (The grain boundary effect in Fig 22 is due to chromate passivation of the cadmium).

Removal of the cadmium in 30% ammonium nitrate solution, to expose the steel surface, revealed that the bubbled areas were characterized by a surface texture markedly different to that of the unbubbled surface, as shown in Fig 23. More detailed examination showed that corrosion pits were present within each area of the steel associated with bubbling of the cadmium plate, as shown in Fig. 24. This scanning electron micrograph also indicates that the grain boundaries of the austempered steel were well delineated as if by acid attack.

Although the grain boundary attack was present over all the steel surface, it was particularly marked at the bubbled areas.

3.9.3 Conclusion

Hydrogen gassing occurred during plating, leading to a porous cadmium deposit associated with bubbling of the plate at one location.

Excessive gassing was probably initiated at the etched grain boundaries of the steel in general, and at the pitted zones associated with bubbling of the plate in particular.

It is likely that an acid cleaning process and/or insufficient washing, prior to plating, was the main cause of the gassing problem.

The fact that pitting of the steel and bubbling of the plate only occurred at one end of the spring may indicate that droplets of acid solution were in contact with the steel surface for the longest time at this location.

3.9.4 Recommendations

Spring steels can be cleaned using anodic or alkaline techniques as specified in DEF 03-2 for example. Where possible, acid cleaning should be avoided. If an acid pickle is unavoidable, however, pickling should be carried out at room temperature in an inhibited hydrochloric acid solution not exceeding 20% concentration.

3.10 Pitted cadmium plate

3.10.1 Plating problem

The samples were made from flat spring steel strip. The yellow passivated cadmium plate showed extensive pitting of the deposit.

The pitting was much more pronounced on one side of the component, and was particularly severe at a concave recessed area at one end of the spring.

3.10.2 Detailed examination

The sample was examined on the scanning electron microscope.

The general nature of the pitted cadmium surface is shown in Fig 25. Numerous shallow pits were present, in sizes varying from 0.01mm to 0.1mm diameter. More detailed examination showed that small particles of debris were often present at the base of the pits, as shown in Figs 26 and 27.

Selective removal of the cadmium in 30% ammonium nitrate solution revealed a well prepared steel surface with no evidence of pitting or severe grain boundary attack, as shown in Fig. 28.

A combined light treatment of ultrasonic cleaning in ammonium nitrate solution removed the debris from the base of the pits in the cadmium plate and revealed the position previously occupied by debris at the bottom of the pit, as shown in Figs 29 and 30.

3.10.3 Conclusion

Pitting of the cadmium plate occurred as a result of debris particles which effectively settled onto the surface of the spring during electroplating. This effect can occur as a result of dust/anode sludge etc. held in suspension in the plating bath.

3.10.4 Recommendation

Pitting of the deposit can be avoided by more effective filtration of the bath. More effective process control during electroplating can avoid this effect.

3.11 "Spotting" of cadmium plate

3.11.1 Plating problem

The samples consisted of compression springs made from spring steel wire. The springs were cadmium plated without subsequent chromate passivation. The components appeared satisfactory immediately after plating, but a brown "spotting" effect developed in storage over a period of time.

3.11.2 Detailed examination

Scanning electron microscopy revealed clear evidence of the "spotting" effect, as shown in Fig. 31. More detailed examination clearly showed small crevices in granular/porous cadmium deposits at the centre of each spotted zone, as shown in Fig. 32. Further examination confirmed that both the granularity and the porosity of the cadmium plate was present over most of the surface.

3.11.3 Conclusion

The granular and porous cadmium deposit retained cyanide solution in the crevices after the final washing operation. During storage, the retained cyanide seeped out onto the cadmium surface to give the observed brown "spotting" effect.

It is likely that the porous cadmium was due to a high plating current and/or a plating bath which was slightly out of balance during electroplating.

3.11.4 Recommendation

The "spotting" effect can be prevented by thorough washing of the plated spring, followed by chromate passivation. However the effect can be eliminated at source by better process control during electroplating.

4. DISCUSSION

Examination of the eleven case histories gives the following breakdown with respect to the source of electroplating problems investigated at SRAMA.

Cause of problem	% of samples
1. Incorrect specification	20
2. Incorrect pre-cleaning	20
3. Inadequate control during plating	60

4.1 Incorrect specification

Correct specification is especially important for nickel and chromium electroplating, since both these metals have relatively poor throwing power during deposition.

Thus nickel will tend to give thinner deposits in recessed areas, and hence the thickness of the plate should be controlled with reference to concave or recessed locations.

Similarly, chromium has little or no levelling action to "smooth out" shallow marks etc on wire or strip surfaces. However small surface marks can be smoothed by bright nickel plating using a bath of good levelling action, after which a thin flash of bright chromium will give the cosmetic finish required. Full details of the nickel and chromium electroplated finishes can be found in the current edition of BS 1224.[1]

4.2 Incorrect Pre-cleaning

Selection of the correct pre-cleaning procedures is crucial for high strength spring materials. Springs will often be operating at high stresses and can fail in service as a result of corrosion pitting introduced during pre-cleaning operations in addition to the gassing/blistering problems introduced during electroplating of pitted material.

Cleaning procedures suitable for high strength steels are fully documented in Defence Standard 03-2.[2] The recommended procedures essentially consist of organic, alkaline and anodic cleaning processes, although restricted use of inhibited dilute hydrochloric acid solutions are also permitted as part of a full cleaning cycle.

4.3 Inadequate Control During Plating

Whilst caution is necessary in drawing specific conclusions from the limited number of samples examined, it seems clear that inadequate process control during electroplating may be a major source of the plating problems experienced by the SRAMA membership. Consequently most of the plating problems would be eliminated by exercise of better process and quality control procedures during plating.

Thus, where plating work is contracted out, good quality finishes should be obtained by using metal finishing firms which are registered under the BSI Quality Assurance Schedule (QAS) 3138/20. This schedule amplifies the requirements of BS 5750: Part 2 in relation to metal finishing processes, and should therefore ensure that components are correctly plated to specified requirements.[3] However, de-embrittlement treatments appropriate to the springs should be clearly stipulated, since most of the volume electroplating business involves low strength materials rather than the high strength steels used in springmaking.[4]

Full details of suggested de-embrittlement procedures are available in recent SRAMA reports.[5,6]

5. CONCLUSIONS

1. Examination of electroplated springs, representing typical plating defects, suggests that the majority of plating problems are associated with poor process control during electroplating.

2. The work has indicated that correct specification of electroplated finish and use of appropriate pre-cleaning procedures can play a significant role in reducing electroplating problems.

3. Scanning electron microscopy has been shown to be an effective technique for assessing the quality of electroplated deposits.

6. REFERENCES

1. BS 1224:1970 "Electroplated coatings of nickel and chromium".

2. Defence Standard 03-2/1:1970 "cleaning and preparation of metal surfaces".

3. B.S.I. Buyers Guide, 1985. Contains details of UK companies offering services to appropriate Quality Assurance Schemes, including QAS 3138/20 for metal finishing. Available free of charge from:

    Certification and Assessment Service,
    British Standards Institution,
    P.O. Box 375,
    Milton Keynes MK14 6LO.
    Tel: 0908 315555

4. Defence Standard 03-4/1:1977. "The pre-treatment and protection of steel parts of specified maximum tensile strength exceeding 1450N/mm^2."

5. Reynolds, L.F., "Research at SRAMA into hydrogen embrittlement of zinc electroplated carbon spring steel." Springs, Vol. 24 No. 2, October 1985, pp 19-31.

6. Reynolds, L.F., "Upon the factors affecting delayed failure in electroplated carbon spring steels." SRAMA Report 393, May 1986.

7. BIBLIOGRAPHY

Theoretical

The following two books give a good introduction to the theoretical basis for electroplating. A working knowledge of electro-chemistry is required.

1. West, J.M., "Electrodeposition and corrosion processes." Pub. Van Nostrand Reinnhold Ltd., 1971.

2. Fraunhofer, I.A., "Basic Metal Finishing." Pub. Paul Elek (Scientific Books) Ltd., 1976.

Practical

Practical guidance for electroplating can be found in the following publications.

1. "Canning Handbook" (on Electroplating) Pub. W. Canning Limited, 1982. Available from E. & F. N. Spon Ltd., Tel. 01-583-9855.

2. Graham, A.K., (Ed)., "Electroplating Engineering Handbook." Pub. Van Nostrand Reinhold Ltd., 1971.

3. "Guide to Nickel Plating." Inco. Alloys International Ltd.

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10. Geduld, H., "Practical problems in bright cadmium barrel plating." Ibid., Sept. 1964, pp. 64-7.

11. Siddall, M., "Converting from cadmium to zinc plating", Products Finishing, Aug. 1964, pp. 50-65.

TABLE 1 Electroplated finishes examined and reasons for rejection of plated steel samples

Sample No.*	SRAMA Project Ref. No.	Spring type	Electro-plate	Complaint
1	552/5B	Flat clips	Barrel plated Nickel	Blistering/poor adhesion
2	552/2	Conical	Vat plated Nickel	Dull/pitted deposit
3	552/5P	Flat clips	Barrel plated Nickel	Brown "spotting" of deposit
4	552/8	Wireform+	Vat plated Chromium	Pitted deposit
5	552/7	Compression	Barrel plated Chromium	Dull areas on bright chrome
6	552/9A	Compression	Barrel plated Zinc	Rough deposit
7	552/9B	Compression	Barrel plated Zinc	Dull deposit
8	552/9C	Compression	Barrel plated Zinc	Blistered deposit
9	552/6	Flat clip	Barrel plated Cadmium	Blistered deposit
10	552/10	Flat clip	Barrel plated Cadmium	Pitted deposit
11	552/3	Compression	Barrel plated Cadmium	"Spotting" of deposit.

* Identification No. with reference to Section 3 of report.
\+ Mild steel.

Scanning Electron Micrographs

Sample 1: Nickel plate

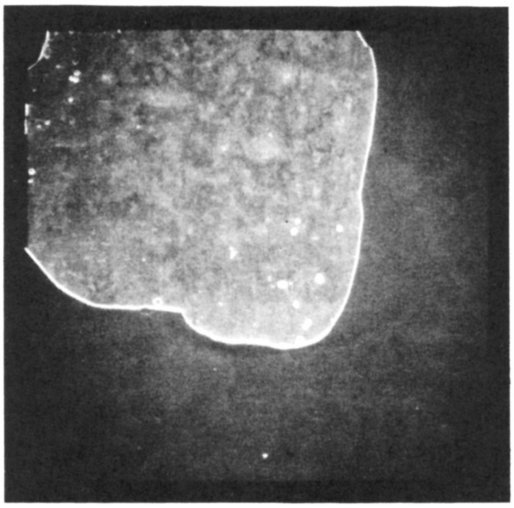


Fig 1 X 110

General view of corrosion pits at steel surface underneath nickel blister

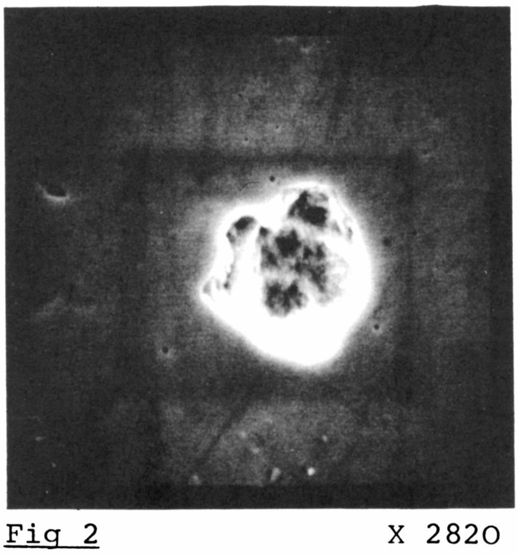


Fig 2 X 2820

Detail of corrosion pit at steel surface underneath nickel blister

Scanning Electron Micrograph

Sample 2: Nickel plate

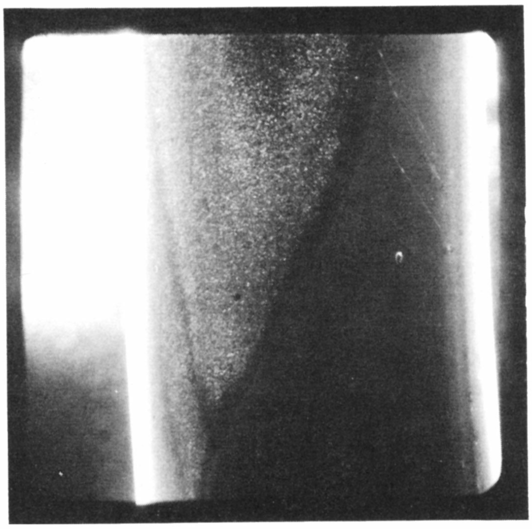
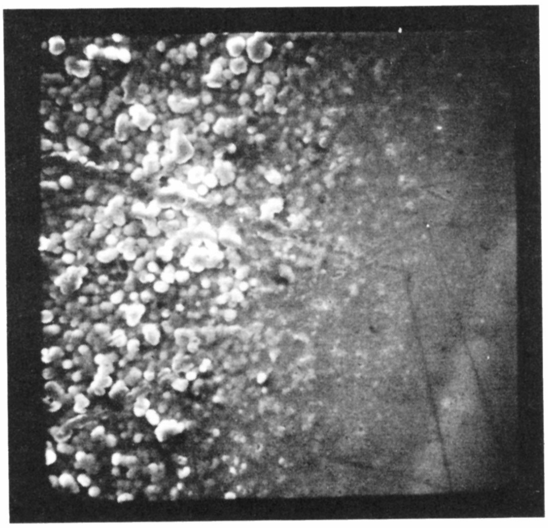


Fig 3 X 70

View of dull area on bright nickel plated surface.

Dull nickel



Bright nickel

Fig 4 X 1440

Detail of adjacent dull/ bright nickel areas

Sample 2: Nickel plate

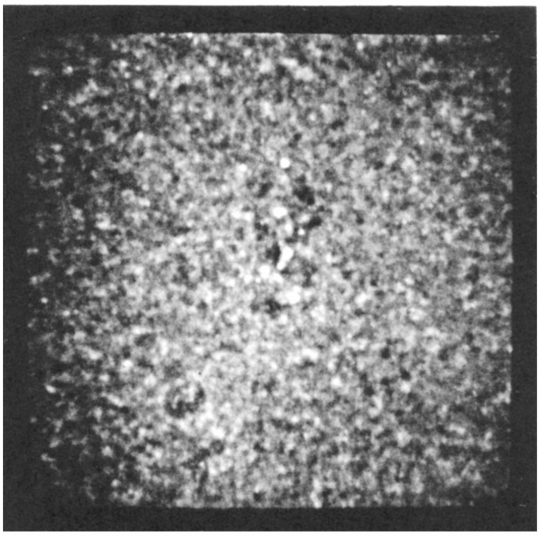


Fig 5 X 2760

Detail of brown/purple discoloured area on nickel plated surface.

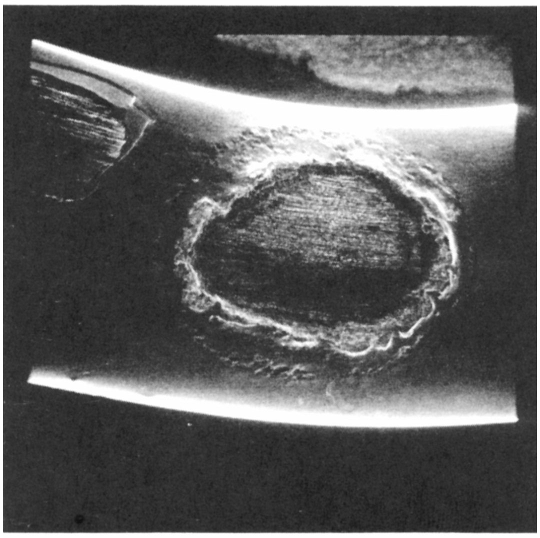


Fig 6 X 60

Unplated area which was associated with red/brown greasy deposit.

Sample 3: Nickel plate

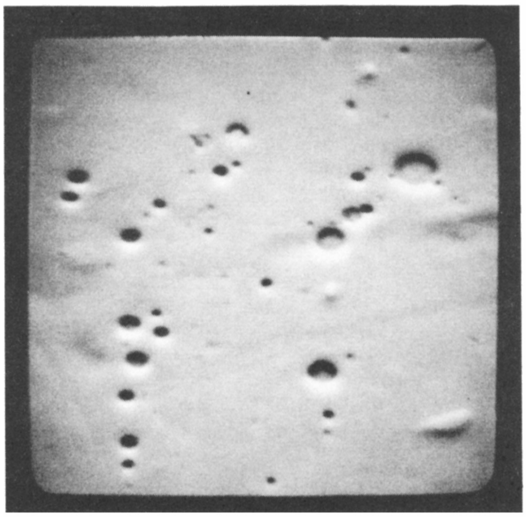


Fig 7 X 5100

Detail of pits in nickel plate at area of thin deposit

Sample 4: chromium plate

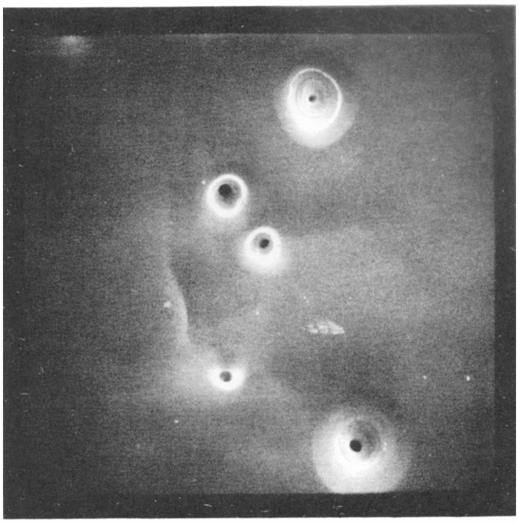


Fig 8 X 145

General view of pits in chromium plate

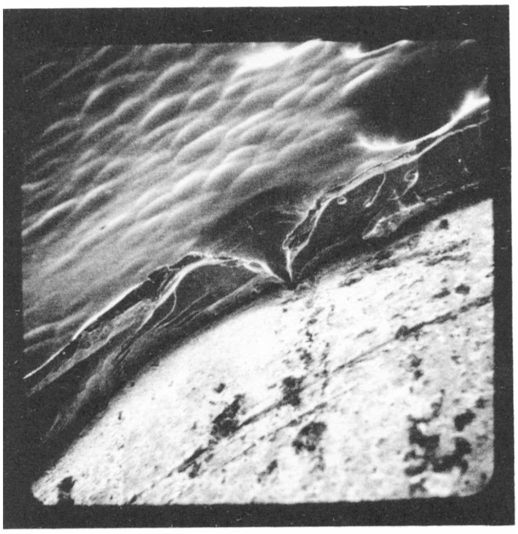


Fig 9 X 130

View of pit at area of fractured chromium plate

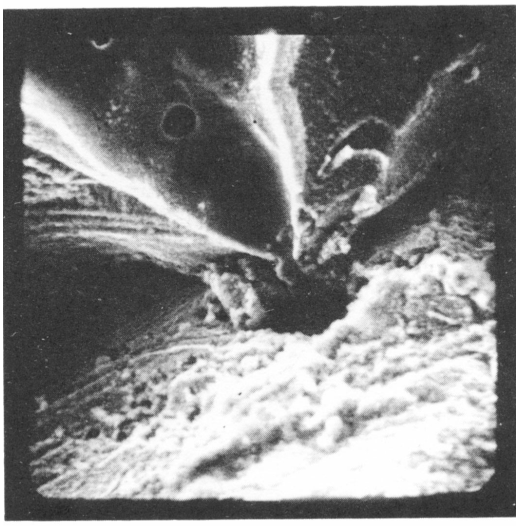


Fig 10 X 1320

Initiation of pit in chromium plate at intrusion on steel surface

Optical Photomicrograph

Sample 4: chromium plate

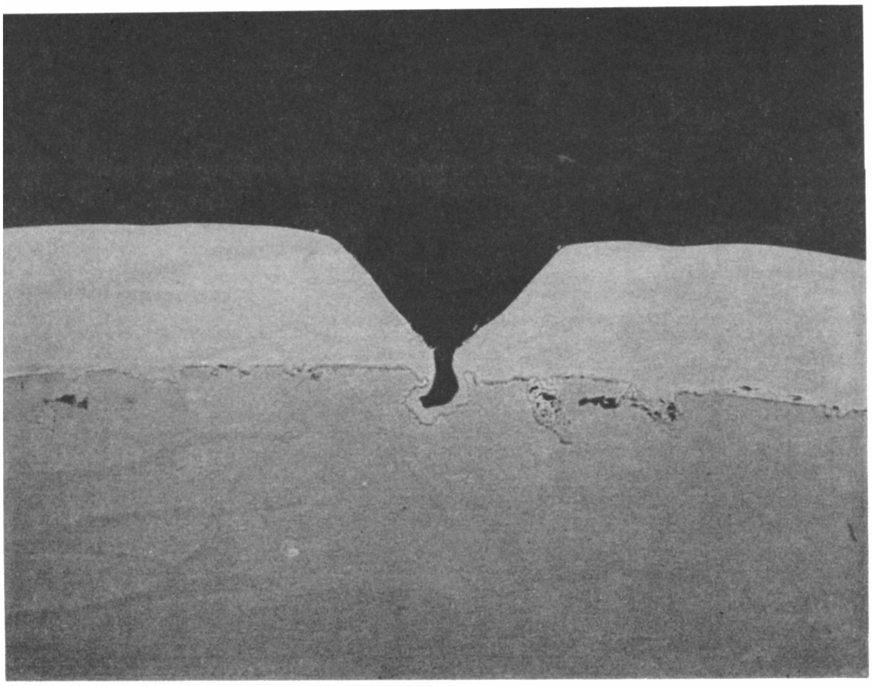


Fig 11 X 205

Section through pit in chromium plate, showing initiation of pits at intrusion in steel surface

Scanning Electron Micrograph

Sample 5: chromium plate

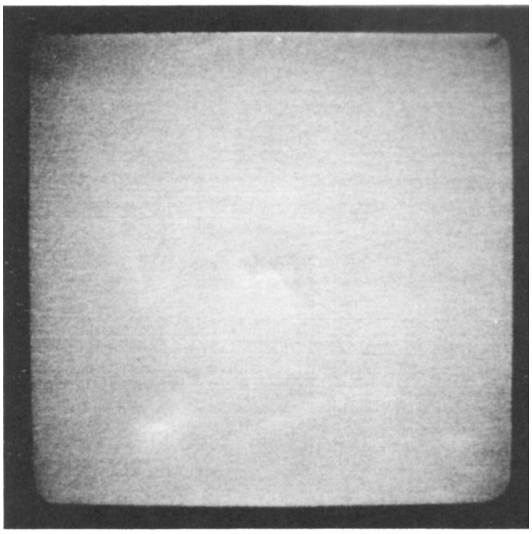


Fig 12 X 310

General view of bright chromium plate at close coiled area of spring

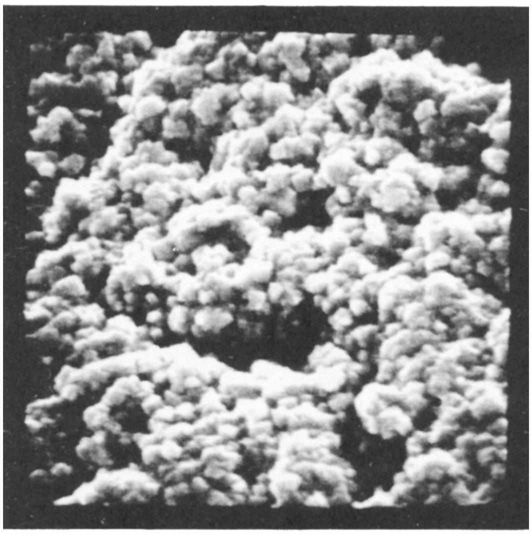


Fig 13 X3200

Detail of burned chromium plate at open coiled area of spring.

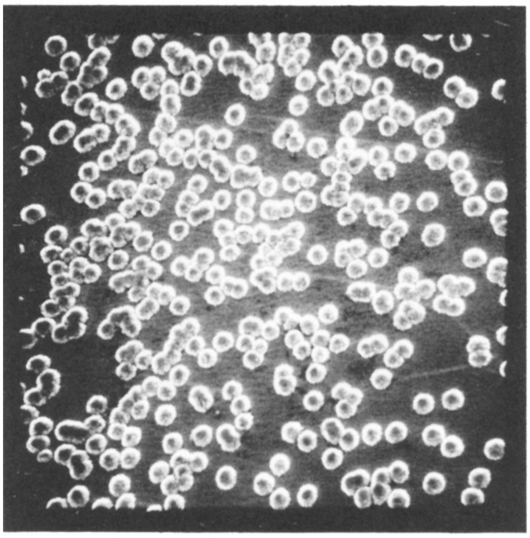


Fig 14 X 330

Transition zone of bright/ burned chromium plate at open coiled area of spring

Sample 6: Zinc plate

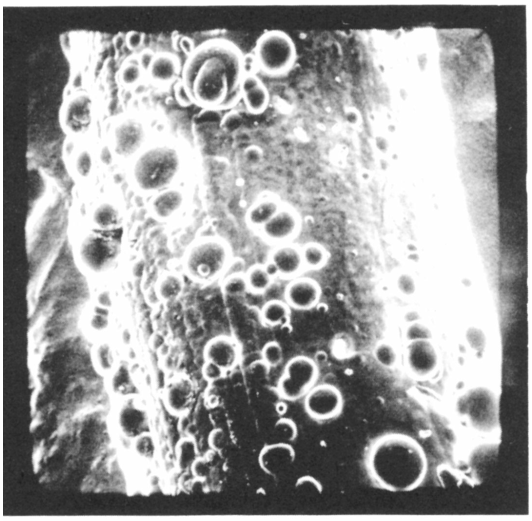


Fig 15 X 30

General appearance of granular plate

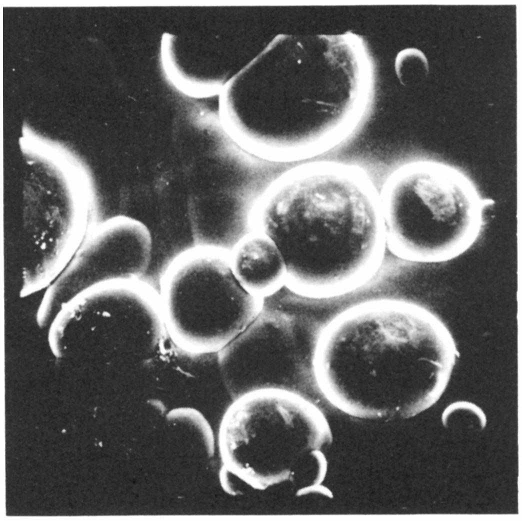


Fig 16 X 125

Detail of granular plate

Sample 7: Zinc plate

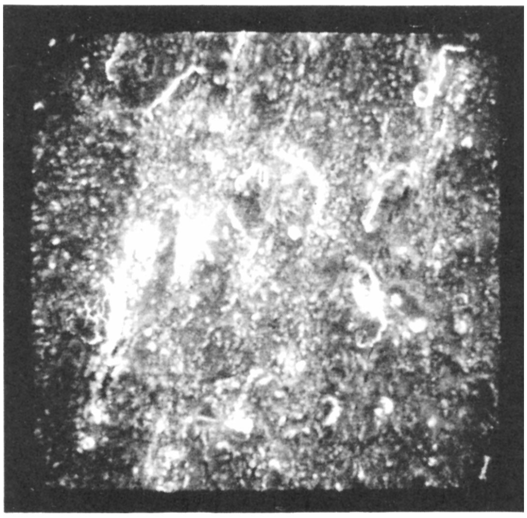


Fig 17 X 600

General appearance of rough plate

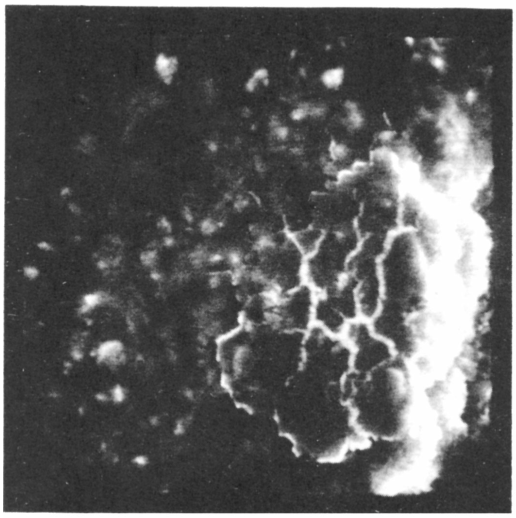


Fig 18 X 3000

Detail of rough plate showing nodular growth.

Sample 8: Zinc plate

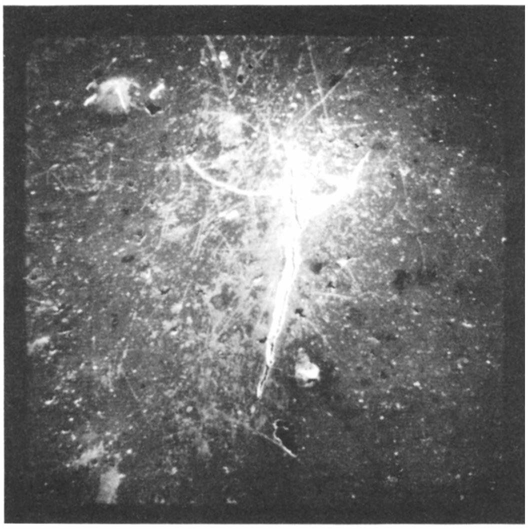


Fig 19 X 110

General appearance of low adhesion plate

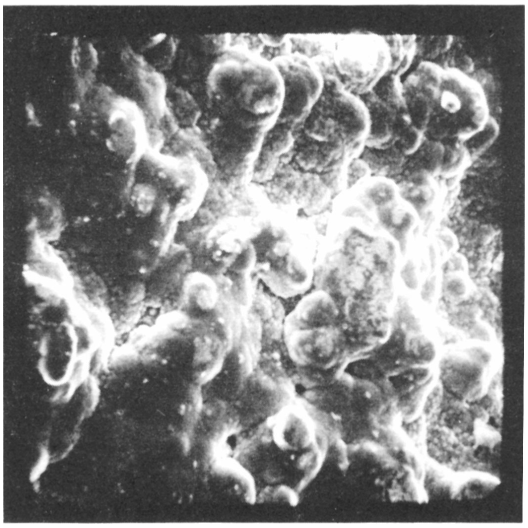


Fig 20 X 600

Detail of nodular deposit on low adhesion plate.

Sample 9: Cadmium plate

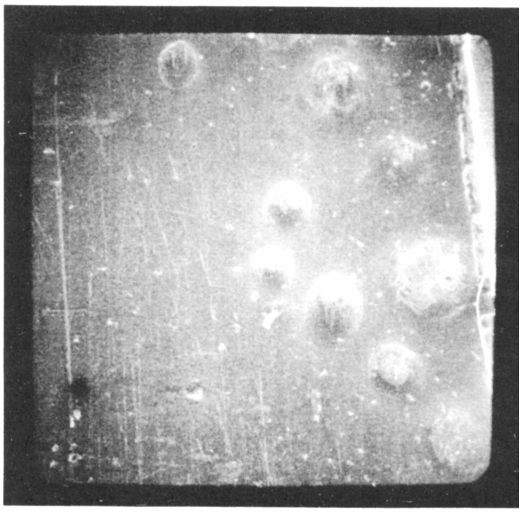


Fig 21 X 25

General view of blistered cadmium plate

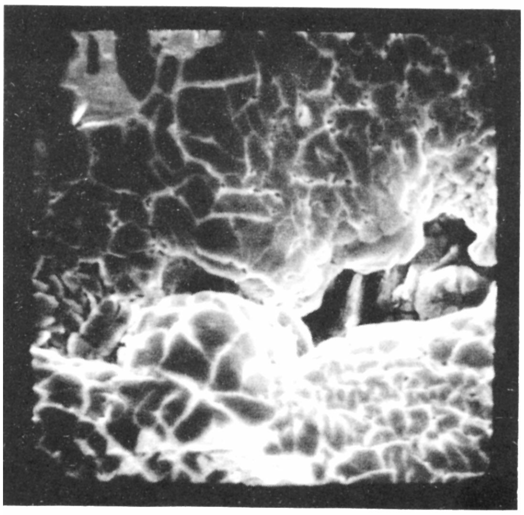


Fig 22 X 1080

Detail of porous cadmium plate at blistered zone.

Sample 9: steel surface after cadmium plate removed

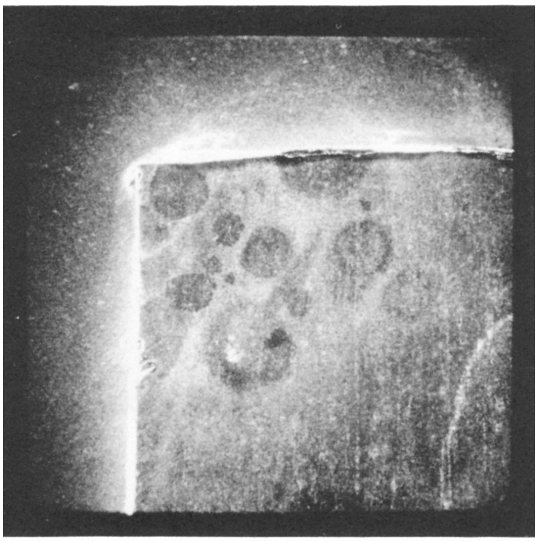


Fig 23 X 25

General appearance of steel surface at blistered area

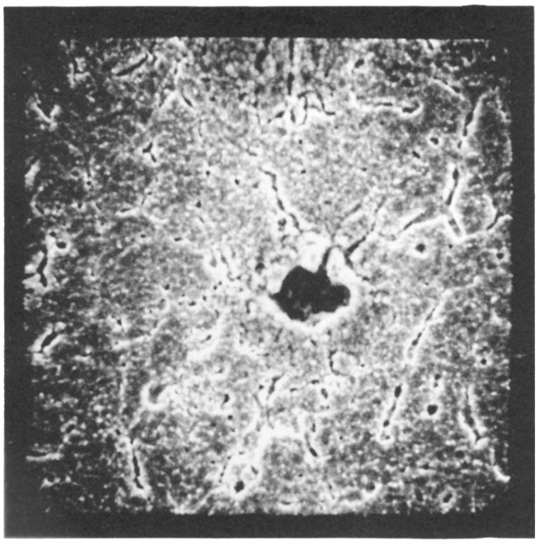


Fig 24 X 1020

Detail of corrosion pit and grain boundary attack on steel surface at blistered area.

Sample 10: Cadmium plate

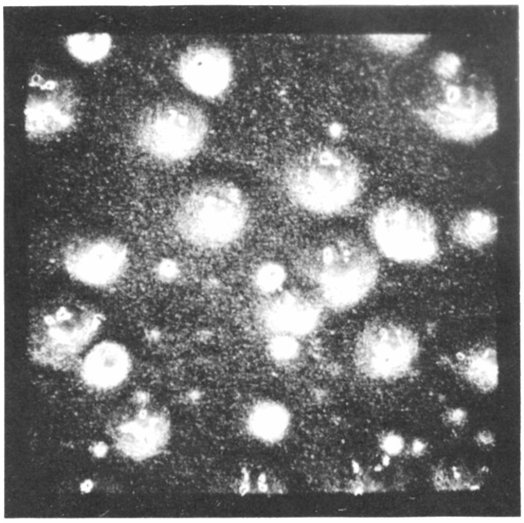


Fig 25 X 110

General appearance of numerous pits in cadmium plate

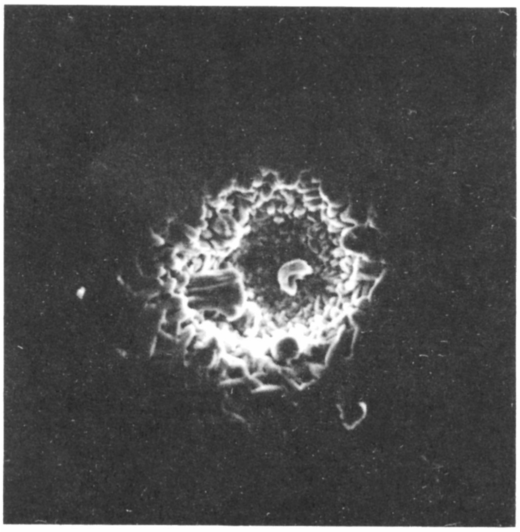


Fig 26 X 1080

Typical pit in cadmium plate

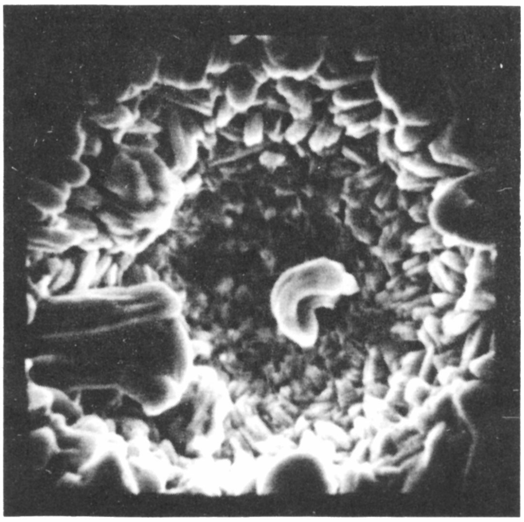


Fig 27 X 2640

Detail of pit showing debris particle which initiated defect.

Sample 10: Surface after selective removal of cadmium plate

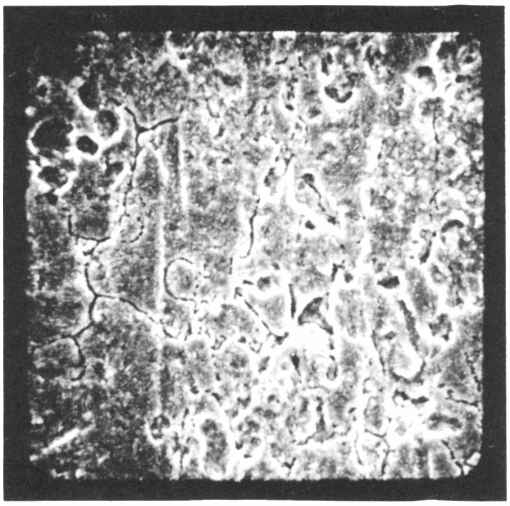


Fig 28 X 1080

Satisfactory steel surface revealed after cadmium plate removed.

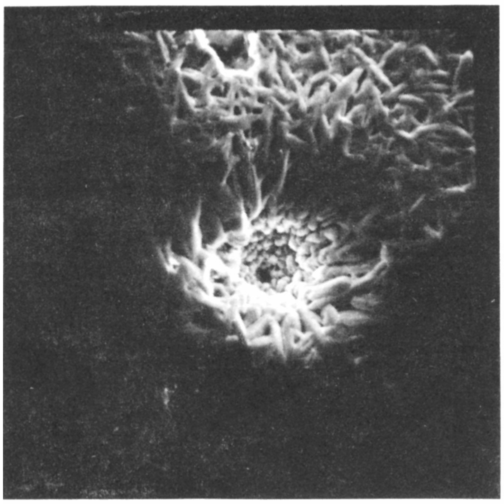


Fig 29 X 1080

Typical pit in cadmium plate after debris particle removed.

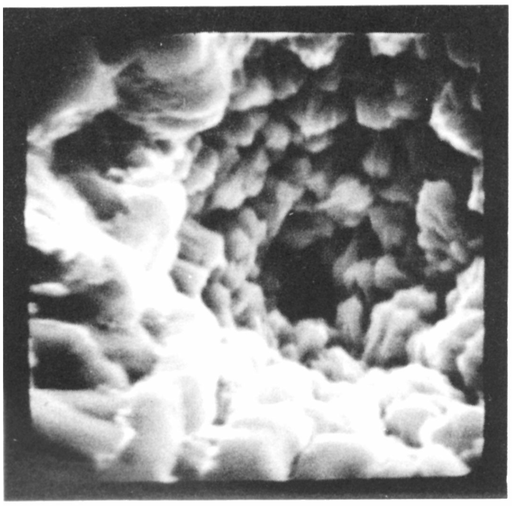


Fig 30 X 5400

Detail of bottom of pit in cadmium plate, showing position previously occupied by debris particle which initiated defect.

Sample 11: Cadmium plate

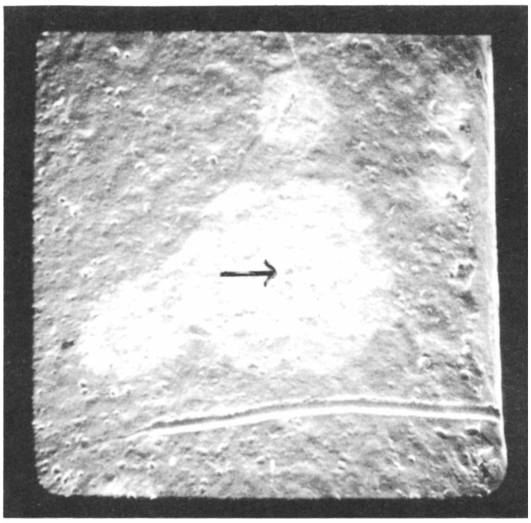


Fig 31 X 33

Area of brown spotting stain on cadmium plate

Fig 32 X 515

Detail of granular deposit at centre of spotted zone above (arrowed). Note also porosity of plate.