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ABSTRACT

A study has been made of the effect of different metallographic mounting and polishing techniques on the appearance in cross section of the oxide layers formed on zirconium and its alloys under various conditions of oxidation.

Optical micrographs were prepared after the various stages of polishing and compared with scanning electron micrographs of oxide fracture surfaces.

For post-transition oxides on Zircaloy-2, similar detail was visible both on the fracture surfaces, and on polished surfaces after prolonged vibratory polishing. It is concluded that surfaces prepared by this technique probably best represent the 'as-grown' structure of the oxide.

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CRACKS; CROSS SECTIONS; ELECTRON MICROSCOPY; FRACTURES; INTERFACES; LAYERS; METALLOGRAPHY; OPTICAL MICROSCOPY; OXIDATION; POLISHING; SURFACES; ZIRCALOY; ZIRCONIUM; ZIRCONIUM OXIDES

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1. INTRODUCTION

Metallographic techniques are frequently used in assessing the corrosion behaviour of the zirconium alloys used for cladding fuel elements and as structural components in water-cooled reactors. The amounts of corrosion product oxide and the associated precipitates of hydride which accumulate during a given exposure can be determined from measurements made on the metallographic cross section. Measurements of the oxide thickness can be made for films as thin as one µm (Daniel et al. 1962) if suitable mounting and polishing methods are used.

A variety of techniques can be used satisfactorily where the oxide thickness is the only quantity of interest. However, if the finer details of the oxide structure are to be interpreted the technique used is more critical, and interpretation in some instances is still a matter for debate.

It is well known that a severely deformed layer is produced during the grinding and polishing processes used in metallography. Samuels (1967) has discussed the different processes which occur during the grinding, rough polishing, and fine polishing of various materials. Abraded surfaces generally contain a damaged layer produced by a combination of the cutting, indenting, and rolling action of the abrasive particles. If this layer or even parts of it are left in the final surface, it can result in the appearance under the microscope of false structures.

A recent study (Bradhurst and Heuer 1971) was made of the mechanism of the 'transition' from protective to non-protective oxide growth, which occurs for zirconium and its alloys. It was found that the metallographic cross sections of pre-transition and post-transition oxides were quite different in appearance. Pre-transition oxides were relatively homogeneous, whereas the post-transition oxides had a layered structure in which the layers were apparently separated by short irregular cracks. Discussion has arisen from this work as to the structure of the layered post-transition oxide (Cox 1971, Bradhurst and Heuer 1971). In a private communication, Cox claims that the layered structure seen in post-transition oxide does not inherently contain cracks parallel to the metal-oxide interface, but that this type of crack is the result of the mounting and polishing technique used. Using a technique developed by Ells (1968), he has produced polished cross sections of post-transition oxides which apparently do not contain cracks.

It is necessary to establish whether cracks are present in the 'as-grown' oxide to distinguish between the opposing theories of the transition process in zirconium alloys, namely, the 'recrystallisation' mechanism, and the 'mechanical failure' mechanism. The first theory proposes that the stresses arising in the oxide due to the different densities of the metal and oxide are relieved by a recrystallisation process occurring at the transition point. This results in a more porous oxide, which is able to continue growing at an approximately linear rate. The second proposes that stress relief occurs by mechanical failure of the oxide by detachment at the metal-oxide interface. The oxidising medium penetrates along these cracks and a new oxide layer is formed. Repetition of this process at many sites on the metal surface results in an approximately linear oxidation rate.

To help resolve this problem a study was made of the effects of metallographic technique on the appearance of cross sections of oxide films on zirconium and some of its alloys. A number of different mounting and polishing methods were used, and the polished surfaces were compared with fracture surfaces of the same specimen, as seen in the scanning electron microscope. The results show that different metallographic techniques can greatly alter the appearance of the oxide. Some doubt remains as to which technique, if any, gives surfaces which truly represent the as-grown structure of the oxide. However, the results suggest that surfaces prepared by prolonged vibratory Syntron polishing probably best represent the inherent structure of oxides on these alloys.

2. EXPERIMENTAL DETAILS

2.1 Materials

The materials studied were zirconium (crystal bar, reactor grade) Zircaloy-2, and a zirconium-2.5 wt.% niobium alloy, obtained from Ugine Kuhlman Laboratories, France. Maximum levels of impurities present, apart from alloying elements were, in wt.ppm, Al 42; C <200; Ca <30; Cu 34; Fe 350-700; Hf 100-120; N 17-41; O 490-630; P <75; Si 30-38. Prior to oxidation, metal surfaces were wet ground on 600 grade SiC paper, followed by etching in HF/HNO_3 solution (HF : HNO_3 : H_2O in proportions 5 : 45 : 50 by volume) rinsed in hot water for an hour or more, washed in methanol, and finally air dried.

2.2 Oxidation

Samples of these materials were oxidised in various media, including dry oxygen, wet helium (steam), air, and pressurised water, at temperatures in the range 290 - 1150°C. In addition, a post-transition sample of Zircaloy-2, oxidised in 500°C steam, was supplied in the as-polished, and etched condition by B. Cox of Materials Science Branch, CRNL, Canada. This specimen was photographed, remounted, and polished using various techniques, for comparison with other oxides.

2.3 Mounting

Four different mounting techniques were used:

(i) Vacuum impregnation with Araldite

(ii) Vacuum impregnation with Araldite containing a zirconia filler

(iii) Bakelite under pressure

(iv) As for (iii) but with the specimen pre-wrapped and sealed in several layers of 0.0005" nickel foil.

The last technique is similar to that used by Ells (1968) and Cox (1971).

2.4 Preliminary Grinding

After mounting, the specimens were initially ground using silicon carbide abrasive papers with flowing water as lubricant. The specimens were hand-held, and the direction of grinding was changed by 90° once for each paper. The four grades of paper used were 120, 320, 400, and 600.

2.5 Polishing

Three different polishing techniques were used in various combinations following the preliminary grinding:

(i) Diamond polishing with a wheel impregnated with diamond paste

(ii) Diamond polishing using a flat diamond impregnated lap

(iii) Vibratory polishing using a Syntron 11-inch diameter automatic polisher.

Diamond abrasive was used mainly for rough polishing. Specimens were hand-held on a rotating wheel (120 rpm) fitted with a napless nylon cloth impregnated with 4-8 μm diamond paste (kerosene lubricant) and polished for 2-4 minutes. The diamond lap consisted of a 6 μm diamond abrasive embedded in a resin matrix and mounted on an aluminium wheel. The lap resulted in a flatter surface than that produced by the equivalent 'rolling' abrasive, and required polishing times of approximately two minutes.

The Syntron polisher was used in most instances as the final polishing stage, but was also used for some specimens for both rough and final polishing, by eliminating the diamond polishing step. The automatic nature of the Syntron polishing enables a more reproducible appearance to be obtained than do hand-held methods. Specimens were loaded in a 300 g stainless steel 'dead-weight' holder and polished for various times in a slurry of various alumina abrasives in water (Cook, Troughton and Sims polishing compound 1, or 0.1 μm) or an 'attack polish' medium. This consisted of 250 cm^3 of ethylene glycol, 100 cm^3 of the C.T.S. alumina slurry, and 30 cm^3 of a mixture of HF : HNO_3 : H_2O in proportions 8 : 46 : 46 parts by volume.

The attack polish solution was left in the polisher overnight before using it for zirconium specimens, to allow thorough mixing, and for equilibration to occur with the metal components and the nylon cloth. The attack polish produced a flatter surface than could be achieved if the alumina abrasive was used alone. More concentrated solutions than the one described resulted in staining of the metal.

2.6 Photomicrography

Optical micrographs of metallographically polished specimens were produced on a Bausch and Lomb metallurgical microscope, and calibrated optical bench. Interferograms were obtained on a Leitz dual beam interference microscope using either white light or monochromatic light of wave-length 0.55 μm. Scanning electron micrographs were obtained on a JEOLCO JSM-U3 microscope/microprobe analyser.

3. RESULTS AND DISCUSSION

3.1 Post-Transition Oxides

The 'as-received' appearance of the sample of oxide on Zircaloy-2 received from CRNL is shown in Figure 1(a). The specimen was originally prepared by mounting in bakelite using the nickel foil method (see section 2.3), wet ground to 600 grit abrasive, rough polished by hand on a wheel with 3 μm, followed by 1 μm, diamond abrasive, and finally Syntron polished for an unspecified period with a 350 g load, 0.05 μm alumina, and a 'micro-cloth' (B. Cox, Private Communication). The oxide shown in Figure 1(a) has been removed from its mount without damage. The surface has also been etched. This does not cause any noticeable difference in the appearance of the oxide but the resulting deep etching of the metal causes the metal surface to be out of focus in Figure 1(a). The oxide appears to be homogeneous, with surface features of dark contrast distributed more or less evenly across the layer. The interferogram in Figure 1(b) shows that these features are relatively shallow depressions of rounded cross section. Figure 1(c) shows that this appearance is only slightly altered by removing the thin carbon layer by gentle hand polishing on a diamond cloth (the as-received specimen had been examined by S.E.M.)

When the oxide is illuminated in the microscope under polarised light, the outer region of the oxide appears translucent (Figure 1(d)). The texture suggests a layered structure, with the innermost layers appearing in dark contrast.

The specimen was re-mounted by vacuum impregnation in zirconia-filled Araldite, wet ground, and Syntron polished for 17 hours using 1 μm alumina abrasive with no attack medium present. Figure 2(a) shows that the oxide has a layered structure with the short crack-like features parallel to the metal oxide interface. Polishing for a further 16 hours using 0.1 μm alumina abrasive in the attack medium (section 2.5) produced a flatter oxide (Figure 2(b)). The metal oxide interface is now in sharp focus, but otherwise the appearance is similar to Figure 2(a).

The question arises whether the lateral cracks apparent in Figure 2 pre-exist in the as-grown oxide or are produced by the polishing technique. It is helpful in deciding between these alternatives to look at the progressive changes which occur from the rough grinding stage through to the final polish.

3.1.1 Prolonged Syntron polishing

After re-grinding the specimen from its polished state (Figure 3(b)) using 600 grade SiC abrasive paper two areas were selected where the grinding directions were normal, and parallel to the metal oxide interface (Figures 3(a) and 3(a)^1). The changes in appearance of these two areas after Syntron polishing for 1, 3, 5, 8, 24, 96, and 188 hours, are illustrated in Figure 3(b) and 3(b)^1 to 3(h) and 3(h)^1. The interferograms corresponding to the final polished surface are shown in Figures 3(i) and 3(i)^1.

The Syntron polishing action could not possibly be as damaging to the oxide as 600 grade SiC abrasive, and yet no cracks are visible in Figures 3(a) and 3(a)^1. It seems likely therefore that the coarse grinding stage obliterates the fine details by the deformation of a surface layer of material, and the fine structure of the oxide is not revealed until this damaged layer is removed by fine polishing. For example, the large vertical crack in Figures 3(a)^1 to 3(h)^1 (arrow # 1) is better resolved with increasing polishing time. Also an adjacent lateral marking (arrow # 2) is better resolved as polishing progresses. The outer edge of the oxide, and the metal oxide interface including detail of the oxygen-rich zone of the metal are also sharpest in 3(h) and 3(h)^1. On the other hand, some of the smaller lateral markings increase in size, while others become smaller or disappear as the surface is polished away. This would be expected if cracks were present as an inherent part of the oxide structure and were gradually revealed as polishing proceeded. The surface profiles of the features of dark contrast in Figure 3(i) and 3(i)^1 appear to be sharper than those in Figure 1(b), and are consistent with those expected for fine cracks.

The as-received oxide (Figures 1(a) and 1(c)) is similar in appearance to that of Figure 3(c), for which the Syntron polishing time was 3 hours. At this stage of the polishing the damaged layer has almost certainly not been completely removed. The areas showing dark contrast are probably partly polished scratch marks, and larger areas where the coarse abrasive has removed material from the layered structure. Less of this type of damage has occurred in the series $3(a)^1$ - $3(c)^1$ where the coarse grinding was done at right angles to the metal oxide interface.

Prolonged fine polishing (Figures 3(d) and $3(d)^1$ to 3(i) and $3(i)^1$) results in a gradual improvement in the resolution of well-defined features such as the edges, and vertical cracks. It seems reasonable therefore that the resolution of any fine cracks present would also improve.

The changing pattern of fine crack-like markings in Figure 3 is consistent with their being an intrinsic part of the oxide structure. While it could be argued that a structure such as Figure 3(h) or $3(h)^1$ might be produced by the gradual loss of material from a layered structure which does not inherently contain cracks, it is equally likely that coarse grinding together with minimal fine polishing could obscure fine cracks which are actually present in the structure.

3.1.2 Diamond polishing

Figure 4 shows how the appearance of the Zircaloy-2 sample changed when the re-ground surface was polished on a 6 μm diamond lap (Figures 4(a) and $4(a)^1$) followed by polishing with 4-8 μm diamond abrasive on a polishing wheel for 5 and 45 minutes (Figures 4(b) and $4(b)^1$, 4(c) and $4(c)^1$). It is interesting to compare the changes in appearance with increasing time of polishing in Figure 4 with those in Figure 3. The diamond abrasive appears to remove more material from the surface, leading to the rounded type of markings which appear in dark contrast under the optical microscope. Similar markings can be seen on the sample in Figure 1(a), which had also been diamond polished during preparation. Figures 1(c) and $4(b)^1$ have a very similar appearance.

The coarse vertical crack in Figures 4(a) to 4(c) (see arrow # 1) becomes progressively wider with increased time of polishing. The lateral markings which appear as cracks in Figures 4(a) and $4(a)^1$ also broaden progressively to the point where whole layers of oxide appear to have been removed in Figure 4(c) and $4(c)^1$ (arrows # 2). More oxide again has been lost from the surface for the series 4(a) to 4(c) where the initial direction of grinding was parallel to the metal oxide interface.

3.1.3 Mounting technique

To evaluate the effect of the mounting technique, particularly the metal foil/bakelite method, a series of specimens of zirconium-2.5 wt.% niobium alloy, oxidised for various times in oxygen at 450°C, were mounted using methods (1), (2) and (4) (see section 2.3). Figure 5 shows identically polished sections of a post-transition oxide from this series (164 mg dm^{-2}) together with interferograms showing the surface profiles obtained for each type of mount. The oxide mounted in nickel foil (Figure 5(e)) appears to have been firmly supported during polishing (pressure used during mounting was 28.9 MPa (4200 psi)). The interface between the foil and the oxide is sharp, and less rounding of the outer edge of the oxide is apparent in the interferogram (Figure 5(f)) than for the other mounting methods.

However, in other respects the appearance of the oxide cross sections are similar irrespective of the mounting technique. Lateral cracks are apparent, similar to those in the post-transition oxide in Zircaloy-2 (e.g. Figure 3(h)). The very irregular metal oxide interface is characteristic of this alloy and has been shown by Cowgill and Smeltzer (1967) to be associated with the different rates of oxidation of local regions of the alloy which contain different amounts of niobium in solution.

The results in Figure 5 show that the appearance of the oxide is not significantly influenced by the mounting technique. This conclusion is also supported by the fact that a similar appearance to that of the as-received oxide on Zircaloy-2 can be produced without the use of the bakelite/foil technique (compare Figures 1(c) and $4(b)^1$). However, the relatively sharp oxide/mount interface in Figure 4(c) indicates that the foil technique has an advantage over the cold-mounting methods where accurate thickness measurements are required, or for the sectioning of very thin oxide layers.

3.1.4 Fracture surfaces

Examination of oxide layers fractured in tension provides an independent means of deciding whether features observed in the polished cross sections are artefacts or a part of the original structure. The sample used for this study was a post-transition oxide formed on Zircaloy-2 in dry oxygen at 600°C. Figure 6(a) is an optical micrograph showing the oxide in cross section after mounting in Araldite and Syntron polishing for 24 hours. The scanning electron micrographs in Figures 6(b) and 6(c) form a stereo pair and give a more detailed view of the area shown as an inset in Figure 6(a). In the stereo image, the oxide surface slopes away to the right hand side at an angle of about 20°. The dark lines appear to be cracks of various widths and it is possible to resolve some depth in the two largest of these (arrows). Oxide sections similar in appearance to these have also been reported by Dawson et al. (1968).

The corresponding fracture surfaces as seen in the scanning electron microscope are shown in Figures 7 and 8. Figures 7(a) and 7(b), a stereo pair (magnification 68x) show how the specimen was bent, exposing the oxide in cross section. Figures 7(c) and 7(d) (magnification 680x) show how the cracks in the oxide are predominantly parallel to the metal oxide interface at the corner of the specimen. Figure 7(e) and 7(f), a stereo pair (magnification 204x) show an adjacent area of oxide to the left of that shown in 7(c). It is interesting to note that the shape and spacing of the cracks in this stereo view are very similar to those which appear after Syntron polishing, such as in Figure 3(h) or in Figures 6(b) and 6(c).

The micrograph in Figure 8(a) shows an adjacent area of oxide at the same magnification as Figure 7(f). Other areas (see insets) are shown at higher magnification (6800x) in Figures 8(b), 8(c), and 8(d) and (e) (stereo pair). It is clear from these micrographs that the oxide is cracked primarily in a direction parallel to the metal oxide interface. Not all of the cracks are as plainly visible on the fracture faces as those in Figure 8 even at this high magnification. Only those emerging in line with the viewing angle of the scanning electron microscope are readily seen, unlike the polished sections such as Figures 6(b) and 6(c) where all cracks are exposed. The oxides in Figures 8(b) to 8(e) also show evidence of a fine columnar crystallite structure. This has also been observed in pre-transition oxides grown in oxygen at 700°C, and does not therefore appear to be connected with the transition process.

3.2 Other Oxides

Figure 9 illustrates the appearance in cross section of oxides grown on Zircaloy-2 and zirconium under different conditions (indicated). The oxides were mounted either in Araldite or Bakelite, without metal support and were then identically ground and polished, using SiC papers followed by 24 hours of Syntron polishing. The oxides in Figures 9(a) and 9(b) appear relatively homogeneous, whereas the structure of the oxide on Zircaloy-2 after exposure to two different media in Figure 9(c) is obviously duplex. The coarse columnar structure of the oxide grown at high temperature in Figure 9(d) is again clearly different. The differences in appearance between these specimens clearly reflect real differences in the as-grown structure of the oxide layers, and demonstrate that the layered appearance of post-transition oxide is not an artefact associated with the Syntron polishing technique. The duplex structure shown in Figure 9(c) emphasises this point, as the same specimen contains both the layered post-transition oxide as an outer layer, as well as an inner homogeneous oxide layer, characteristic of that grown in oxygenated pressurised water.

4. CONCLUSIONS

(1) The appearance of oxide layers on zirconium alloys can be significantly changed by the metallographic preparation and polishing techniques used.

(2) Prolonged Syntron polishing improves the resolution of details of the cross section such as edges and corners. It seems likely therefore, that the appearance of other fine detail in the oxide with prolonged polishing represents an improvement in the resolution of the inherent structure of the oxide.

(3) This fine detail is lost on re-grinding with coarse SiC abrasive or a 6 μm diamond lap. Some loss of resolution also occurs with 4-8 μm diamond abrasive, probably due to the production of a damaged surface layer.

(4) Comparison of polished sections with fracture surfaces prepared from the same specimen indicates that the Syntron polished surfaces best represent the as-grown structure of the post-transition oxide on zirconium alloys.

(5) Syntron polished sections were of similar appearance irrespective of the mounting method used. The influence of mounting technique appears to be less important than previously suggested (Cox 1971). The bakelite/foil method improves edge retention, and the precision of measurement of oxide thickness.

(6) The as-grown structure of post-transition oxides on Zircaloy-2 probably contains layers separated by cracks. An apparently homogeneous structure can be produced by polishing techniques in which the abrasion-damaged layer is incompletely removed.

5. ACKNOWLEDGEMENTS

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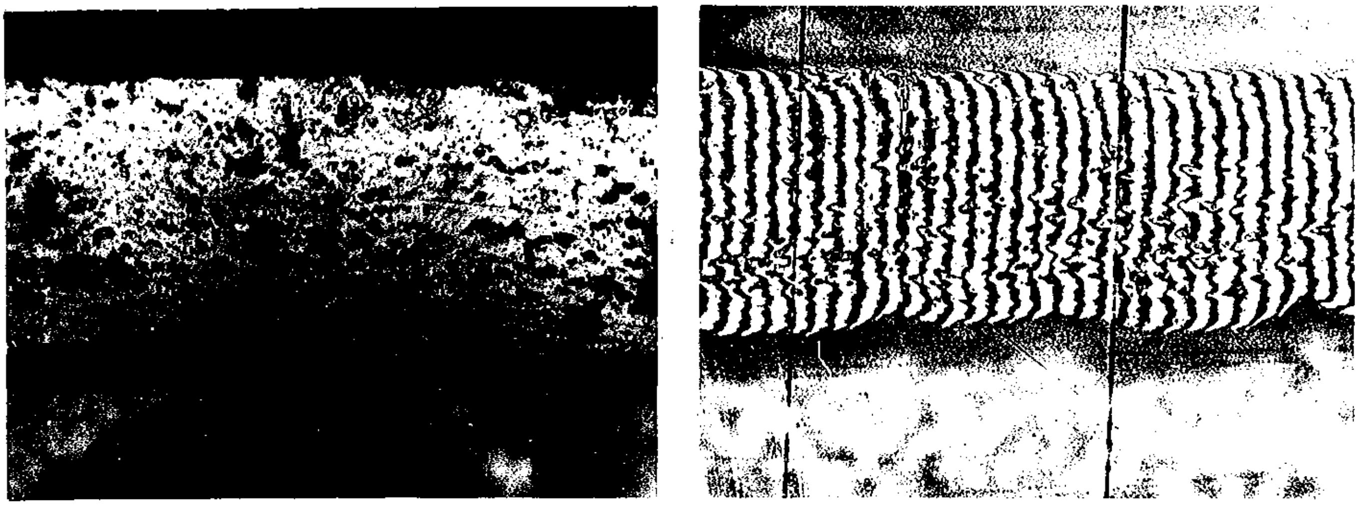
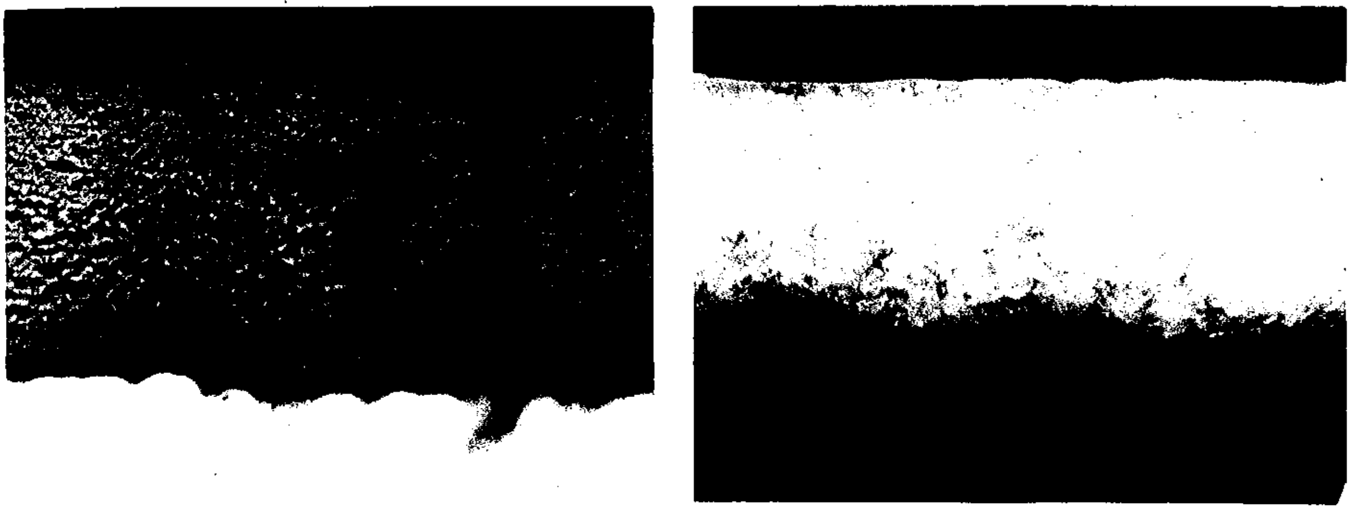


(a) "as-received" appearance

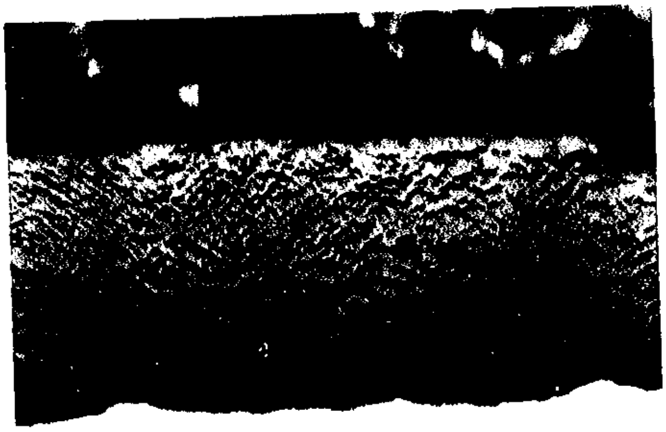
(b) Interferogram (W.L. = 0.55 μm) showing surface profile



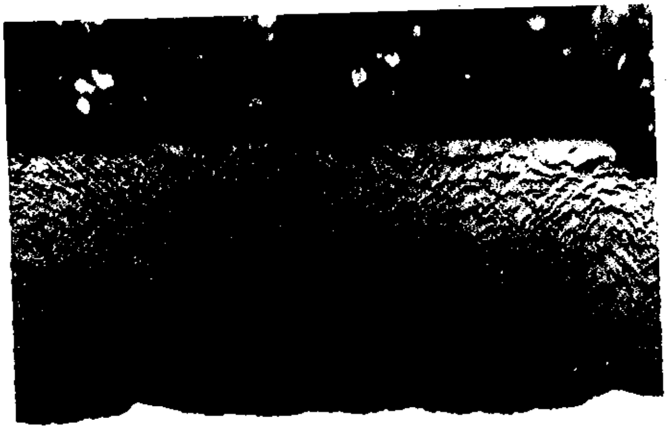
(c) as in (a) after removal of carbon layer

(d) appearance under polarised light

FIGURE 1 POST-TRANSITION OXIDE ON ZIRCALOY-2 (Magnification 425 X)

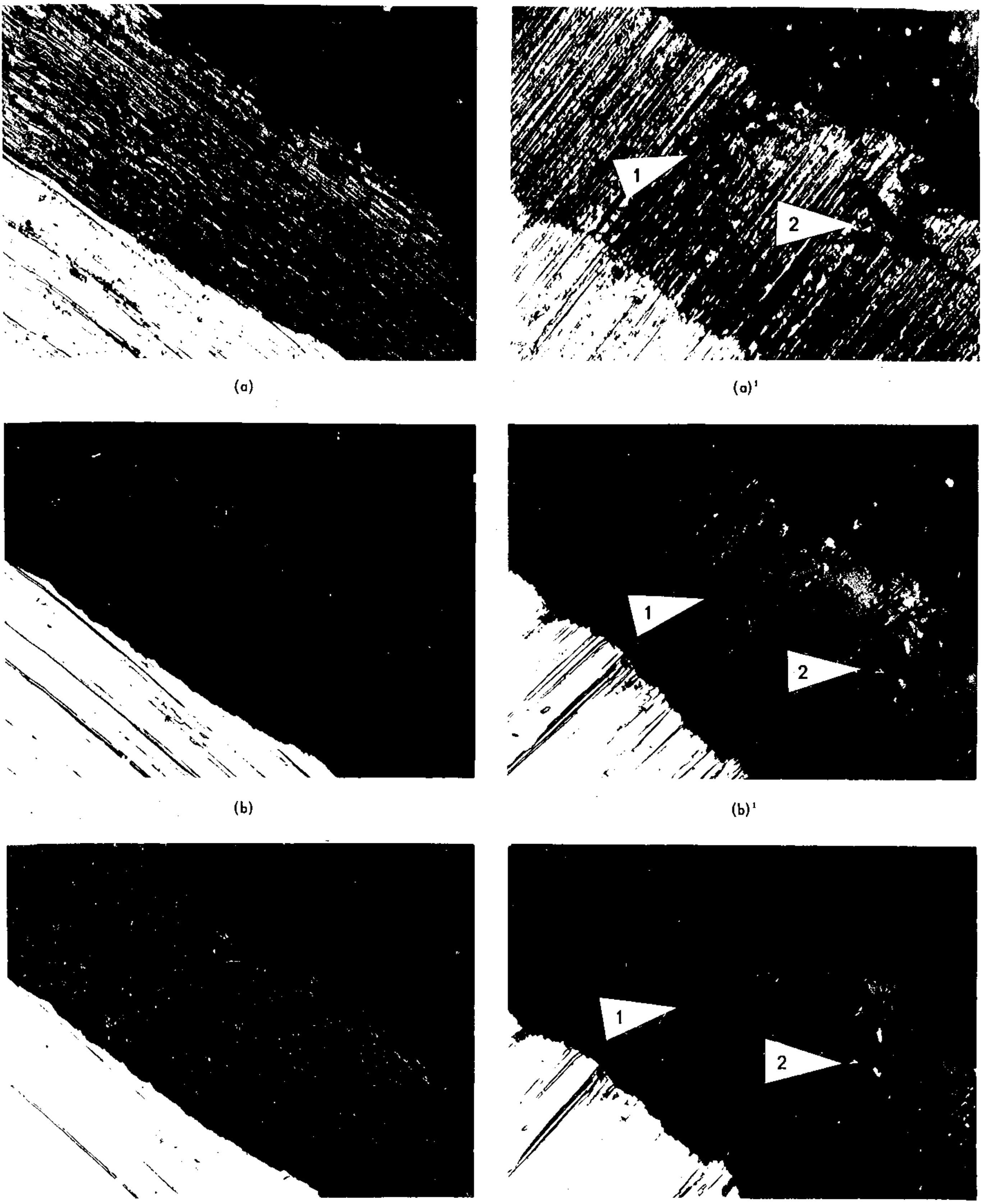


(a) after wet grinding, and 17 hours of Syntron polishing with 1 μm alumina abrasive



(b) after a further 16 hours Syntron polishing with 0.1 μm alumina + an "attack" medium

FIGURE 2 POST-TRANSITION OXIDE ON ZIRCALOY-2 FROM FIGURE 1, REMOUNTED IN ZIRCONIA-FILLED ARALDITE (Magnification 425 X)



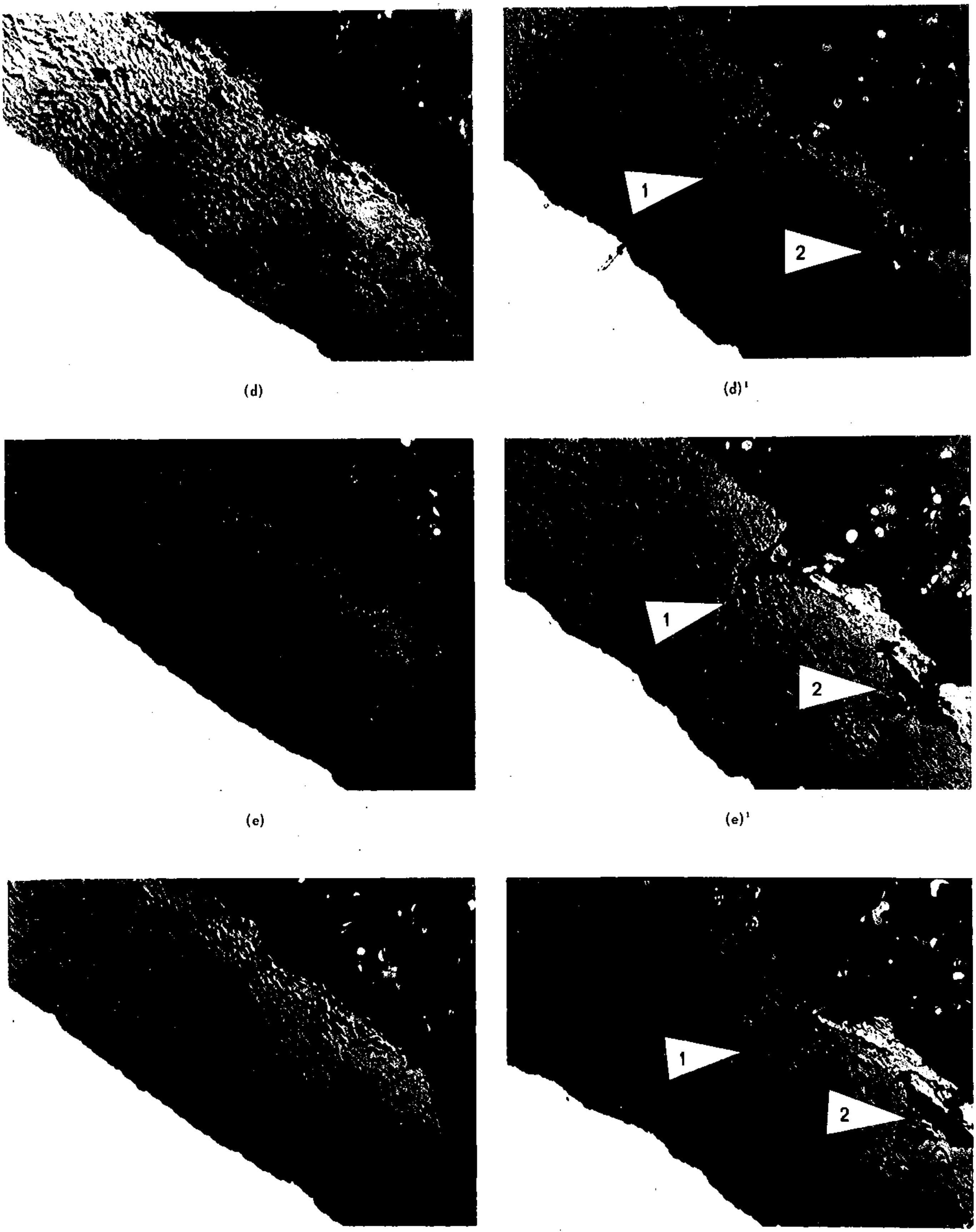
(a) (a)'

(b) (b)'

(c) (c)'

FIGURE 3 POST-TRANSITION OXIDE ON ZIRCALOY-2 REGROUND PARALLEL (SERIES (a) TO (h)) AND NORMAL (SERIES (a)' TO (h)' TO THE METAL-OXIDE INTERFACE (Magnification 425 X). Arrows # 1 and # 2 show improved resolution of detail with increasing polishing time.

(a), (a)' 600 grade SiC paper
(b), (b)' Syntron, 1 m alumina, 1 hour
(c), (c)' Syntron, 1 m alumina, 3 hours

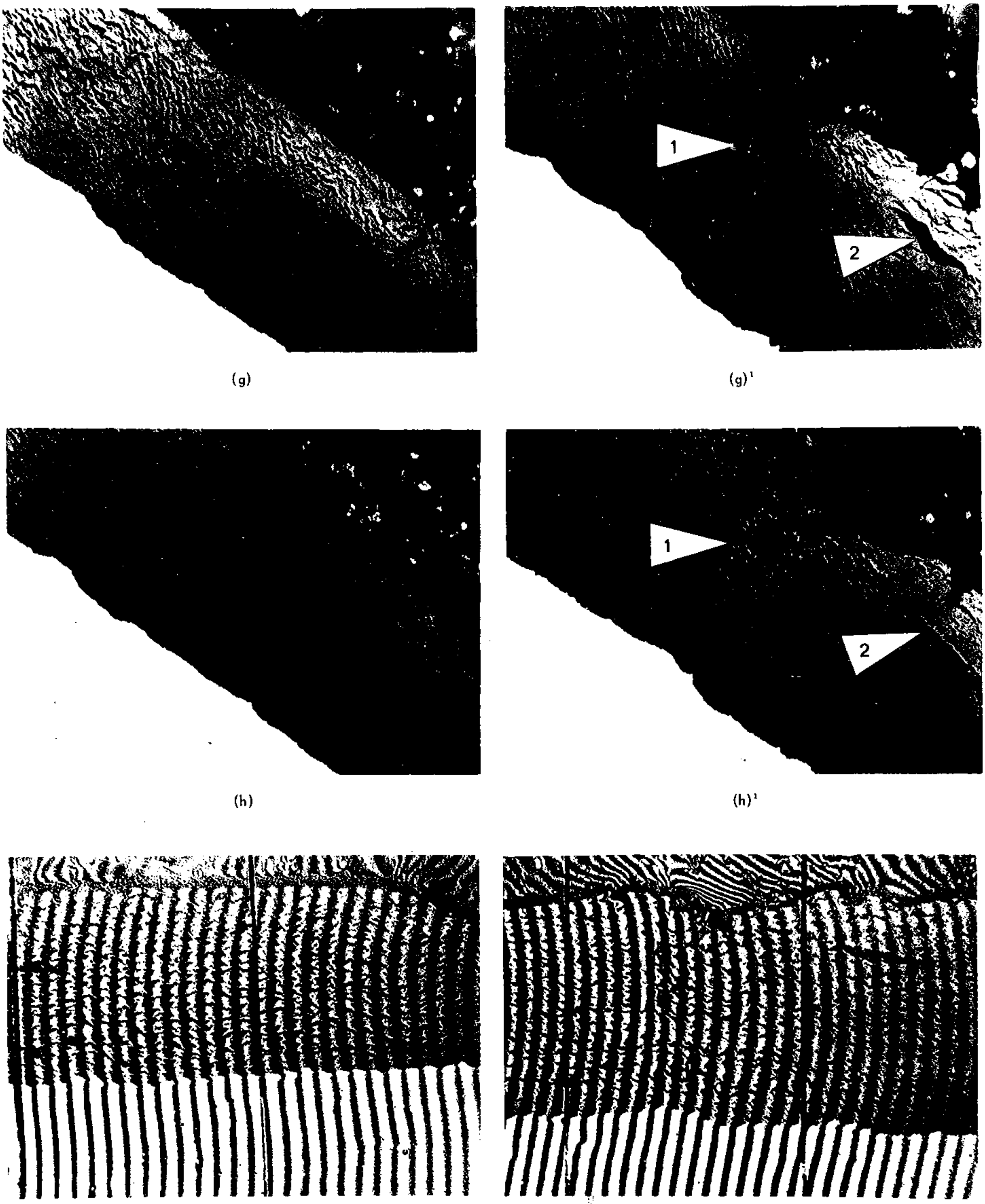


(d) (d)'

(e) (e)'

(f) (f)'

(d), (d)' Syntron, 1 μm alumina, 5 hours
(e), (e)' Syntron, 1 μm alumina, 8 hours
(f), (f)' Syntron, 1 μm alumina, 24 hours

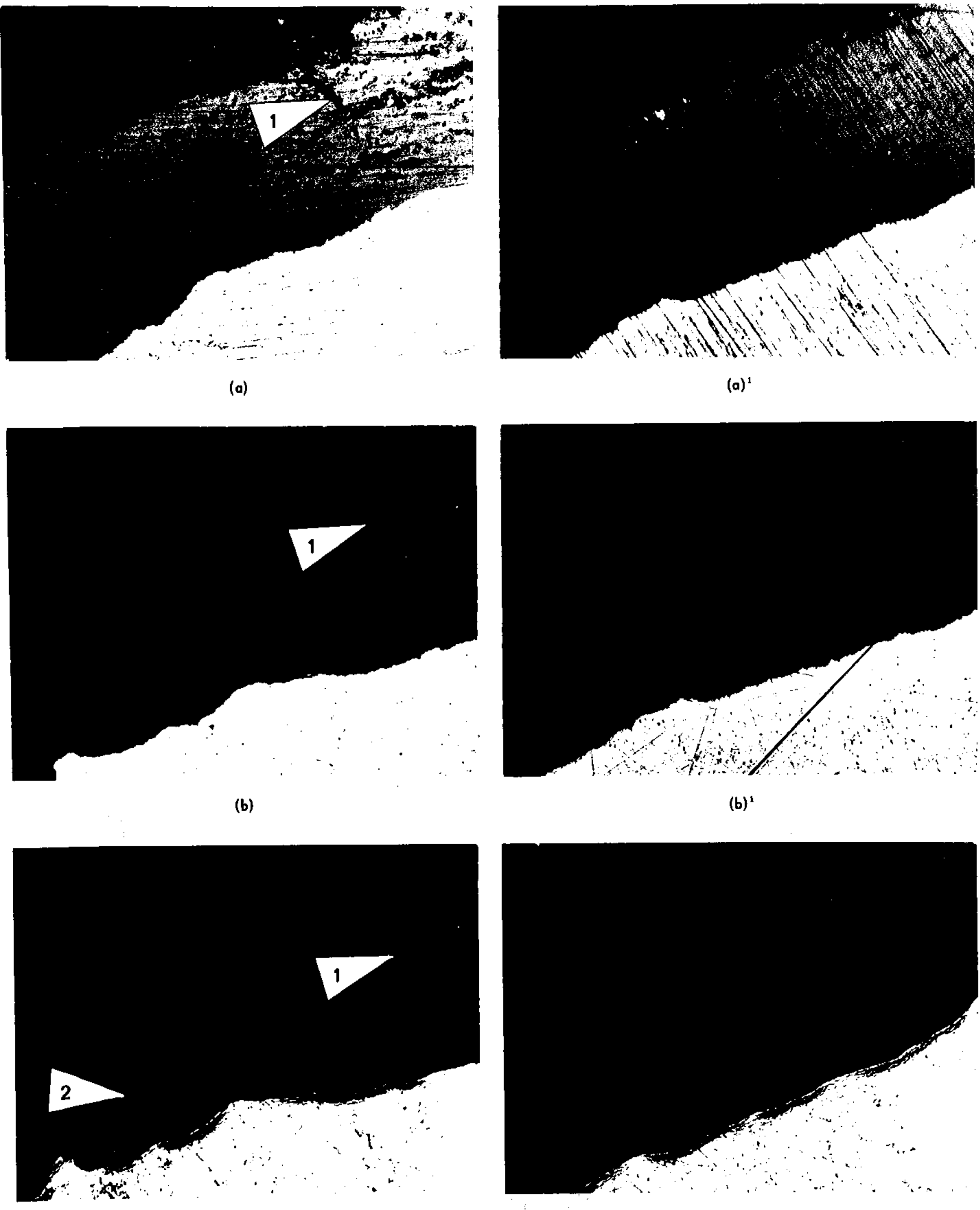


(g) (g)'

(h) (h)'

(i) (i)'

(g), (g)' Syntron, 1 μm alumina, 96 hours
(h), (h)' Syntron, 1 μm alumina, 188 hours
(i), (i)' Interferograms showing surface profiles after 188 hours polishing



(a) (a)'

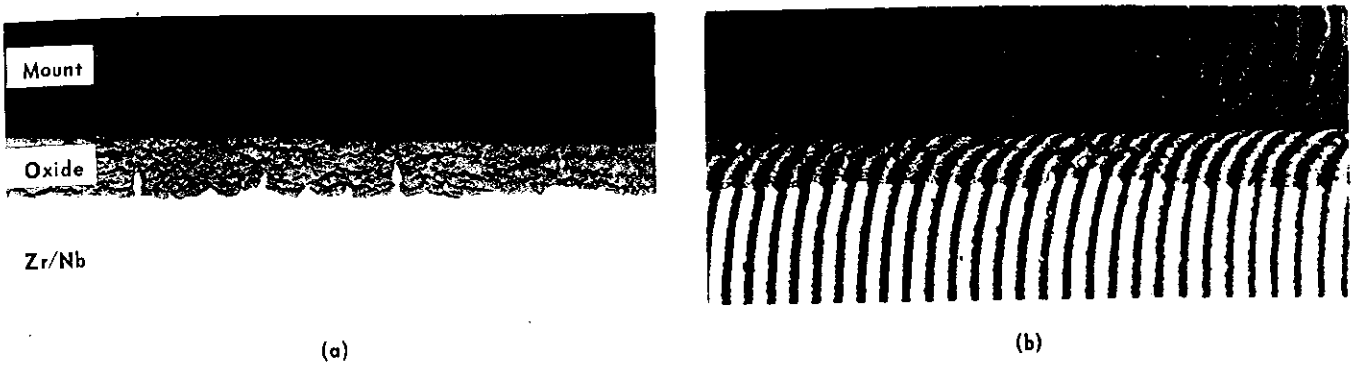
(b) (b)'

(c) (c)'

FIGURE 4 POST-TRANSITION OXIDE ON ZIRCALOY-2 REGROUND AND DIAMOND POLISHED
(Magnification 425 X)

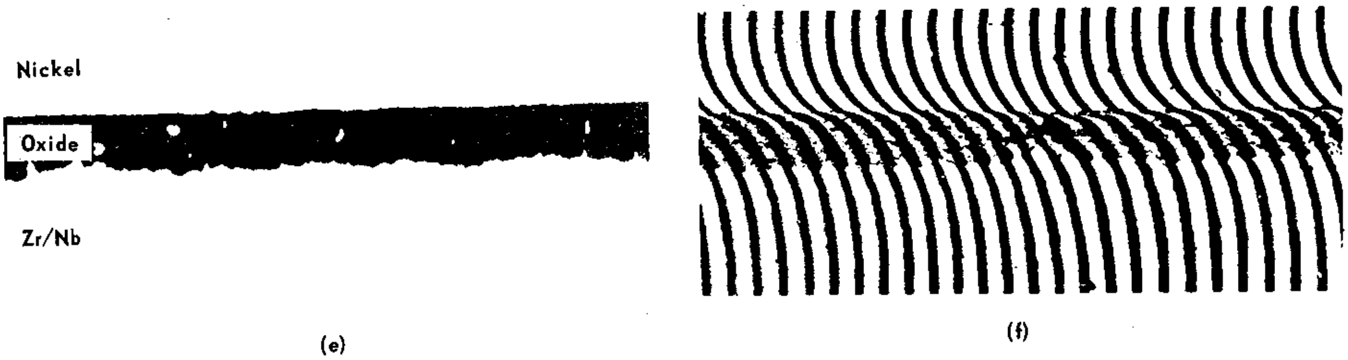
Arrow # 1 shows coarsening of a vertical crack in the oxide
Arrow # 2 shows the apparent loss of a layer of oxide

(a), (a)' after 2 minutes, 6 μm diamond lap
(b), (b)' after 5 minutes, 4–6 μm diamond wheel
(c), (c)' after 45 minutes, 4–6 μm diamond wheel



Vacuum impregnation with Araldite,

Vacuum impregnation with zirconia-filled Araldite



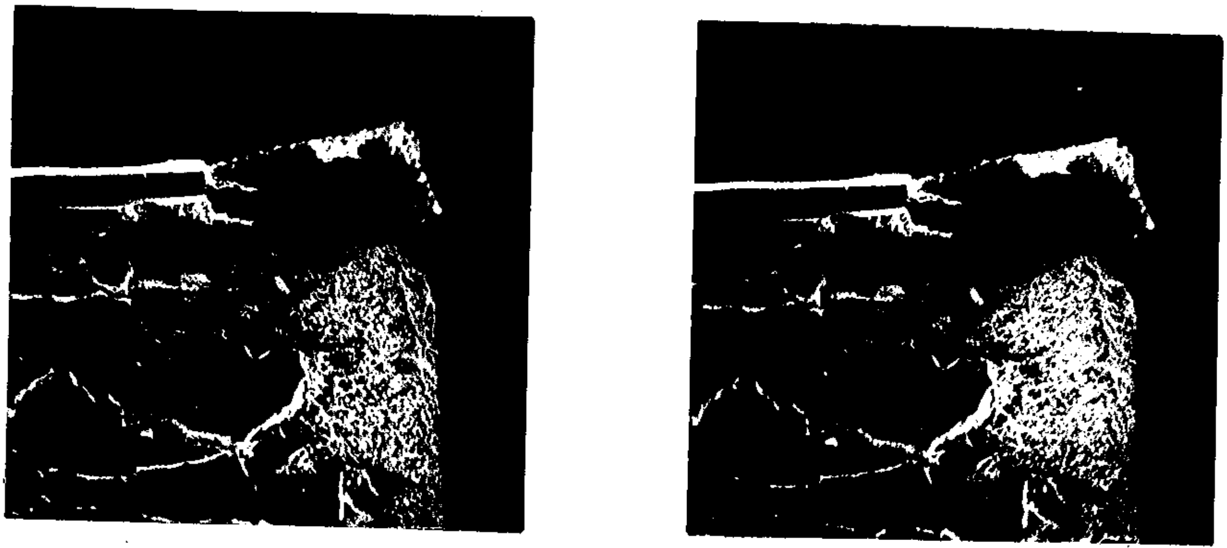
Bakelite/nickel foil, pressure mounted

FIGURE 5 POST-TRANSITION OXIDE ON ZIRCONIUM/2.5 WT.% NIOBIUM ALLOY (164 mg. dm^{-2}) SHOWING THE EFFECT OF DIFFERENT MOUNTING TECHNIQUES ON THE APPEARANCE AND SURFACE PROFILES BY SYNTRON POLISHING (Magnification 425 X)

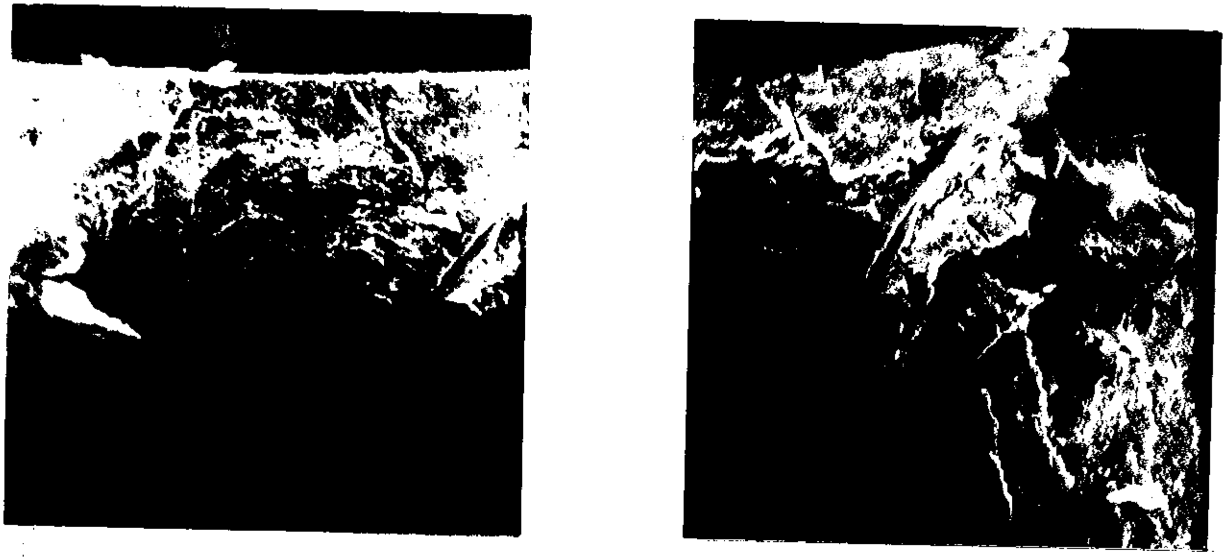
(a) view showing two coarse cracks in the outer layers,
and fine lateral cracks (Magnification 425 X)

(b), (c) stereo pair showing area in the inset of Figure 6 (a) (scanning electron micrographs)
(Magnification 3800 X)

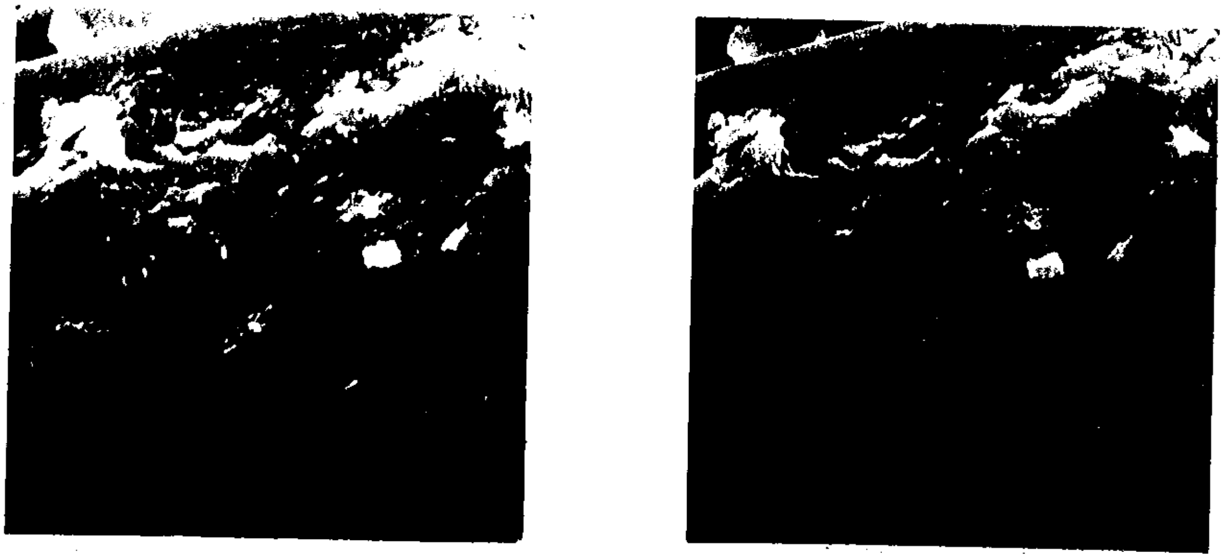
FIGURE 6 POST-TRANSITION OXIDE ON ZIRCALOY-2 PRODUCED IN 600 °C OXYGEN



(a), (b) stereo pair showing how fracture surfaces were obtained by bending the specimen (Magnification 68 X)



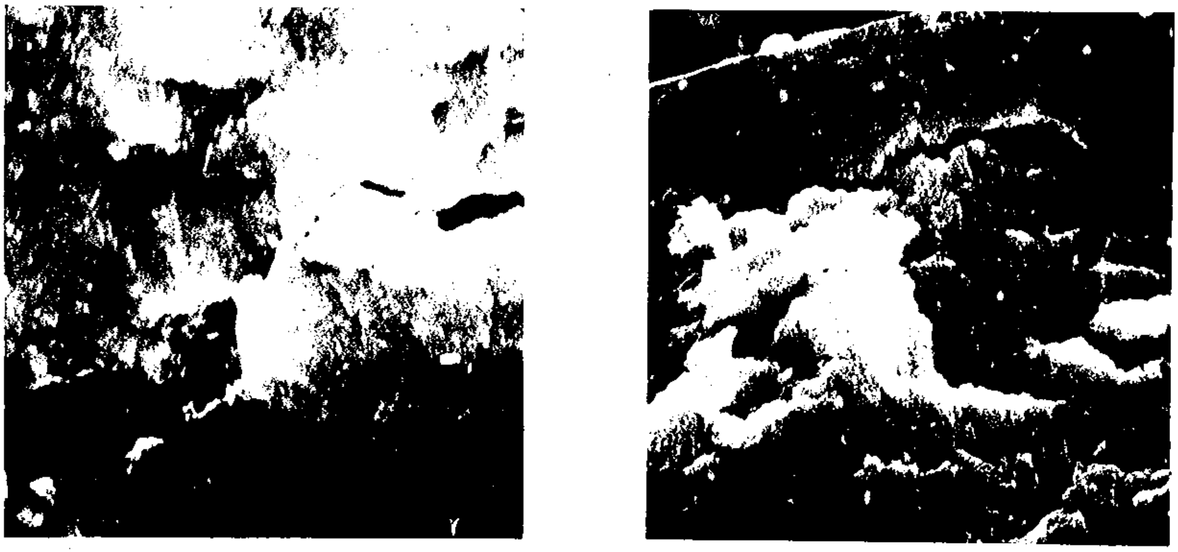
(c), (d) area of oxide near one edge of specimen (Magnification 680 X)



(e), (f) stereo pair showing layered structure and cracks separating the layers (Magnification 2040X)

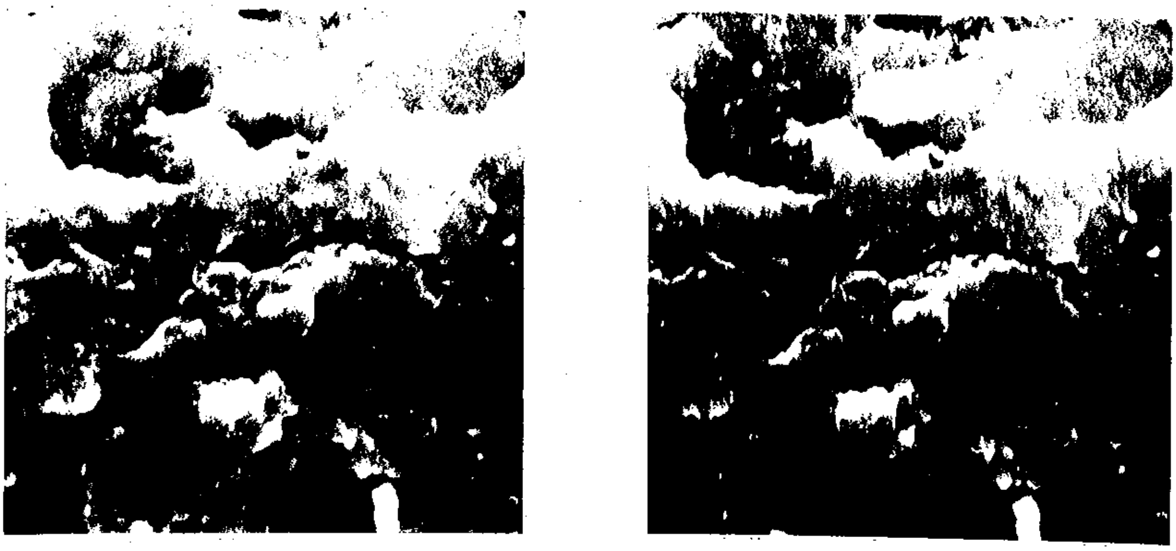
FIGURE 7 SCANNING ELECTRON MICROGRAPHS OF POST-TRANSITION OXIDE FROM FIGURE 6

(a) layered structure (Magnification 2040 X)



(b) enlarged view of inset # 3
(magnification 6800 X)

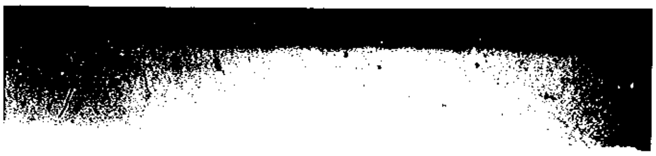
(c) enlarged view of inset # 2
(magnification 6800 X)



(d), (e) stereo pair showing a crack between the oxide layers (inset # 1)
(Magnification 6800 X)

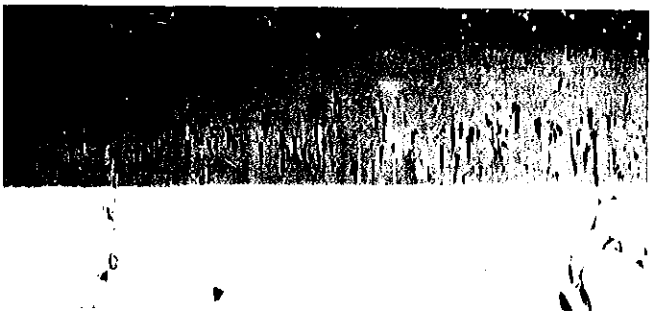
FIGURE 8 SCANNING ELECTRON MICROGRAPHS OF POST-TRANSITION OXIDE ON ZIRCALOY-2

(a) pre-transition oxide on zirconium crystal bar, exposed to dry oxygen at 700 °C, 240 hours (Magnification 425 X)



(b) oxide on reactor grade zirconium exposed to pressurised water and oxygen (27 atm.) in-reactor for 23 days at 290 °C (Magnification 850 X)

(c) duplex oxide on zircaloy-2. Outer layer formed by exposure to wet helium (steam) at 600 °C into post-transition region; inner layer by exposure to oxygenated pressurised water in-reactor for 23 days at 290 °C (Magnification 850 X)



(d) columnar oxide produced by oxidation of reactor grade zirconium in air at 1150 °C (Magnification 85 X)

FIGURE 9 OXIDES ON ZIRCONIUM, AND ZIRCALOY-2 FORMED UNDER DIFFERENT CONDITIONS
SYNTRON POLISHED