Fracture surface examination by selected area electron channelling of single crystals of Mo and 15 at. % Re alloy

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Fracture surfaces of molybdenum single crystals deformed in simple shear between 10^{-3} and 163 sec^{-1} and 148 to 425 K were examined using selected area electron channelling to determine the planes of fracture and to estimate the placticity associated with fracture. These results are correlated with topographic observations and with prior crystal plasticity. Similar, but less extensive, investigation was also made on crystals of molybdenum-15 at. % rhenium. Brittle fracture on several planes besides the {001} is one of the findings; rhenium alloying alters the fracture plane.

1. Introduction

One viewpoint of fracture is that it is the final step in the deformation process. If this view is correct, it should be possible to correlate fracture surface observations with previous deformation history. Although failure analysis has generally shown the validity of this approach, it is very difficult to determine detailed crystallographic and surface deformation information from polycrystalline specimens of engineering materials. Thus, the fracture surfaces of a series of pure molybdenum and molybdenum-15 at. % rhenium single crystals deformed under carefully controlled conditions of strain-rate, temperature and loading mode were examined for their topological details. The elastic and mechanical properties of these crystals, together with the experimental methods employed to measure them, may be found in [1-3].

The technique of selected area electron channelling has been used to determine the crystallography and deformation of observed fracture features. This technique employs the scanning electron microscope used in a special mode* which yields an electron channelling pattern (a diffraction pattern) characteristic of the crystallography of the crystal under study.

2. Experimental

Single crystal rods 3.2 mm in diameter of molybdenum (Mo) and molybdenum-15 at. % rhenium (Mo-15Re) with a ⟨110⟩ axial orientation were produced by the floating-zone, electron-beam technique. The crystals were then held

TABLE I

Material	Specimen no.	Temperature (K)	Strain-rate (sec^{-1})	Fracture stress (kg mm^{-2})	Fracture strain (%)
Mo	1	148	10^{-3}	34	24
	2	148	11	38	9
	3	245	10^{-2}	30	57
	4	245	14	34	12
	5	300	85	49	71
	6	350	150	39	163
	7	425	163	21	217
Mo-15Re	11	125	10^{-1}	34	12
	12	300	155	37	94

*For details on this technique, see [4].

by steel grips and oriented in an X-ray machine so that they could be deformed in simple shear in the ⟨111⟩ direction. A gauge length of 1.8 mm was used. Shearing force was applied using an electrohydraulic testing machine, with the specimen and grips in a temperature controlled gaseous environment. Numerous specimens were tested, with only a few representative of various temperature and strain-rate regimes carried to failure.

3. Mechanical properties

The pertinent mechanical properties of the crystals examined are shown in Table I. A detailed mechanistic analysis of the stress-strain data from these same crystals has been made and may be found in [3].

4. Fracture surface characteristics

Fracture surfaces of the nine specimens listed in Table I were extensively examined in the scanning electron microscope, using both secondary and back-scattered electron modes. Fig. 1 gives a macroscopic view of the specimens examined. Up to 300 K, the surfaces are macroscopically brittle, all showing considerable evidence of cleavage steps and river patterns. Several of the specimens, however, exhibit what might be called conchoidal fracture (non-crystallographic, brittle looking, characteristic of non-metallics, Fig. 1a, c, d and f).

The selected area electron channelling technique was used on many of the observed features to determine the crystal plane of that feature, and to learn something of the plasticity associated with the features examined.

The technique finally evolved in the examination was as follows. The SEM stage was removed to a binocular zoom microscope where the stage controls were used to orient a flat fracture feature of interest perpendicular to the microscope axis. Light incident at ~ 45° was alternately directed on the specimen from two orthogonal directions to help ensure that orientation was perpendicular. Magnifications between × 7 and × 40 were used. The stage was then transferred back to the SEM. Optical axis of the stereo microscope was maintained parallel to the electron-optical axis of the SEM. Channelling conditions were then established, and the feature was examined for orientation and

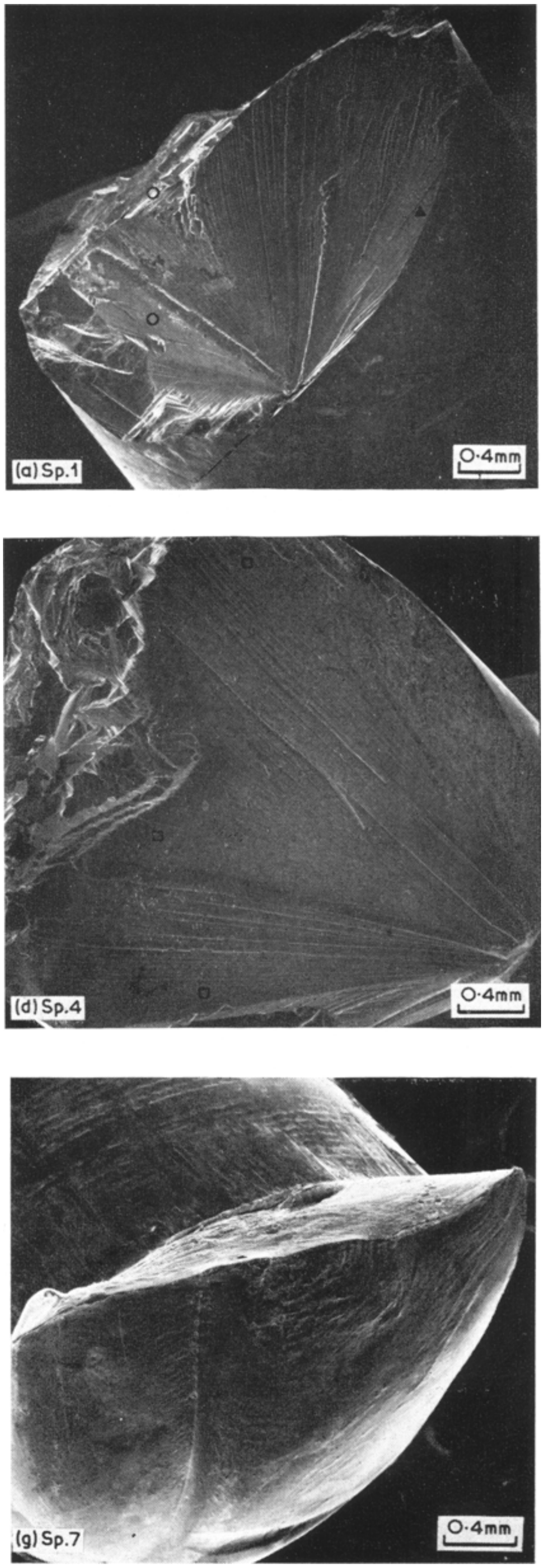


Figure 1

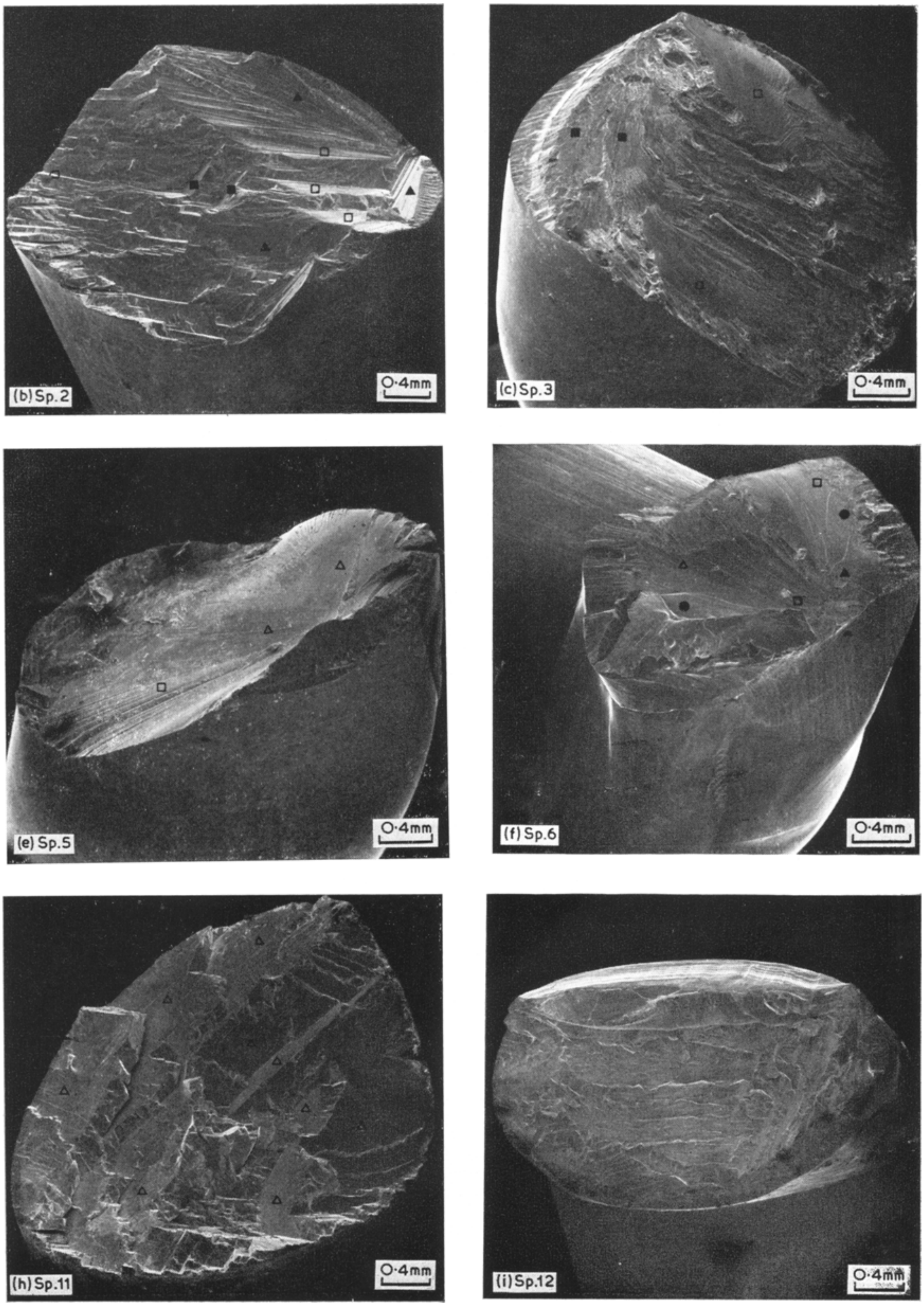


Figure 1 Fracture surfaces examined, showing orientation of the planes as determined. □ = {001}, ■ = {011}, ● = {012}, ○ = {112}, △ = {113}, ▲ = {111}.

deformation. For the SEM used,* it is possible to change the area over which the channelling pattern is taken by various combinations of objective aperture and rocking angle. Interpretation of the channelling pattern becomes more difficult for small included angles, but the larger angles generally result in a larger spot size. An included angle of 13° was used, which gives a spot size approximately 60 μm in diameter.†

Plastic deformation as determined by the electron channelling technique is derived from resolution and contrast changes in the channelling patterns. No entirely suitable technique has yet evolved to quantify these changes, although efforts aimed at better quantification are underway. At present, approximate deformation is being determined by visual comparison with calibrated tensile specimen, and by measurement of resolution of a weak line in the pattern [6]. Crystallographic orientation of the channelling patterns was determined by comparison with a channelling map for molybdenum, shown in Fig. 2.‡ Sufficient deformation existed in several of the specimens to necessitate annealing§ before patterns with sufficient clarity for positive identification could be obtained; annealing precludes further deformation analysis.

The crystallographic orientations shown in Fig. 1 were determined by the technique described above. The occurrence of fracture on planes other than the {001} and {011} is not common, although McNeil and Limb [7] have reported cleavage on {111} and {112} in chromium and on {113} in molybdenum.

Considerable effort was expended to verify the observation of the {001}, {112}, {111}, and {113} planes. In several instances, when one of these high index planes was encountered, the specimen was reoriented to bring a low index pole into view; the angular change necessary was consistent with the original observed direction. Furthermore, removing the stage to the optical microscope further verified that the plane under observation was no longer perpendicular to the optical axis. In several cases, another iteration of alignment and plane identification was performed, yielding the same orientation as previously observed.

Fracture surfaces 1, 5 and 6 are curved, or made up of several nonparallel facets, each having a different orientation. The normals were generally found to lie in the zone whose axis is [220] between the [001] and [111], except on specimen 6, where the curvature was sufficient to include normals near the [012].

For the molybdenum-15 at.% rhenium crystals examined (specimens 11 and 12) no evidence of either {011} or {001} cleavage was found. Specimen 11 showed cleavage on several planes of the {113} family. Specimen 12 is composed of many small features, each indicating relatively large amounts of deformation, and it was not possible to identify the crystallography of any of these features, even after annealing; however, the overall plane of the fracture was identified as the {113}.

An assessment of the deformation associated with the fracture features observed was derived from the quality of the channelling patterns observed,¶ the frequency and coarseness of the steps associated with the cleavage facets, and the edge sharpness of the various features observed. Although difficult to quantify, there existed a general correlation between observed fracture strain and the diffuseness of the observed channelling patterns. This information, taken together with the observations of topographic detail, lead to the general conclusion that the macroscopically observed plasticity is reflected in the plasticity of the fracture features. For example, it was not necessary to anneal specimens

*The selected area electron channelling patterns were generated by an Etec Autoscan which uses the after-lens deflection coil technique.

†Two methods were used which gave approximately the same result: (1) a grain was found having the same diameter as the rocking beam, and (2) the grid method, described in [5].

‡A large backscattered electron channelling map for molybdenum is available on request.

§Annealing was carried out in a vacuum evaporator at 10^{-6} Torr and approximately 600°C for 1 h.

¶Channelling pattern quality is assessed from the resolution of the observed lines in the pattern, angular distortion of the lines, and the contrast of the pattern. Loss of contrast generally accompanies loss in angular resolution for the lines as deformation of the lattice is increased, i.e., the patterns become less clear, or more diffuse, with lattice deformation. At this stage of development of the channelling technique, to obtain quantitative assessment of strain, it is necessary to have a calibration specimen so that pattern quality may be related to known deformation. Pattern quality of the material under study must also be determined prior to deformation. It has been shown [6], however, that 10 to 15% tensile strain is about the maximum strain observable before pattern contrast is totally lost. For the work reported here, only a relative indication of strain is possible since the mechanical tests were performed before development of the channelling technique. Patterns of excellent quality, however, were obtained from untested crystals of the type used for this investigation.

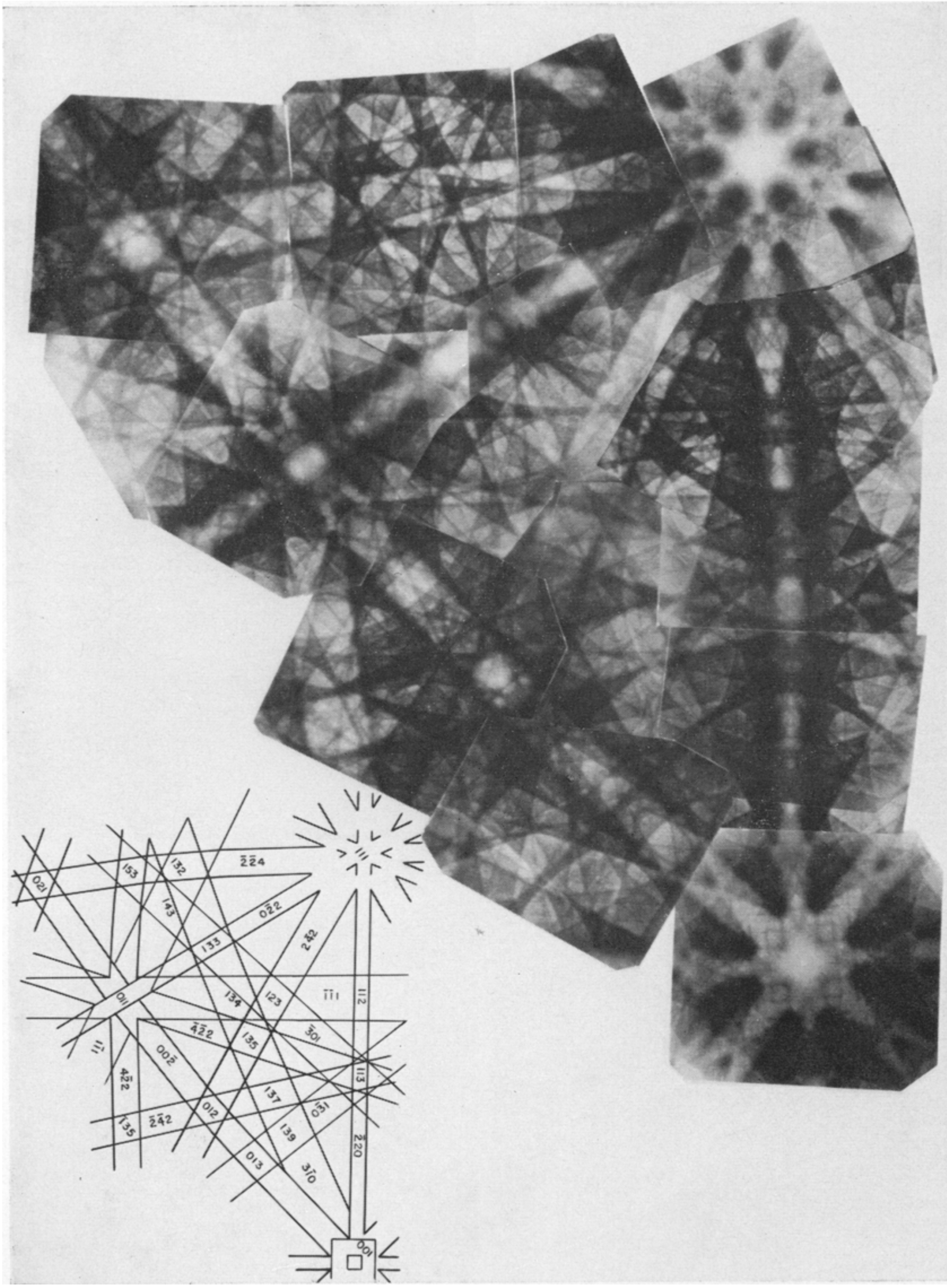


Figure 2 The back-scattered electron channelling map for molybdenum at 20 kV. Orientation of the map as shown in the inset.

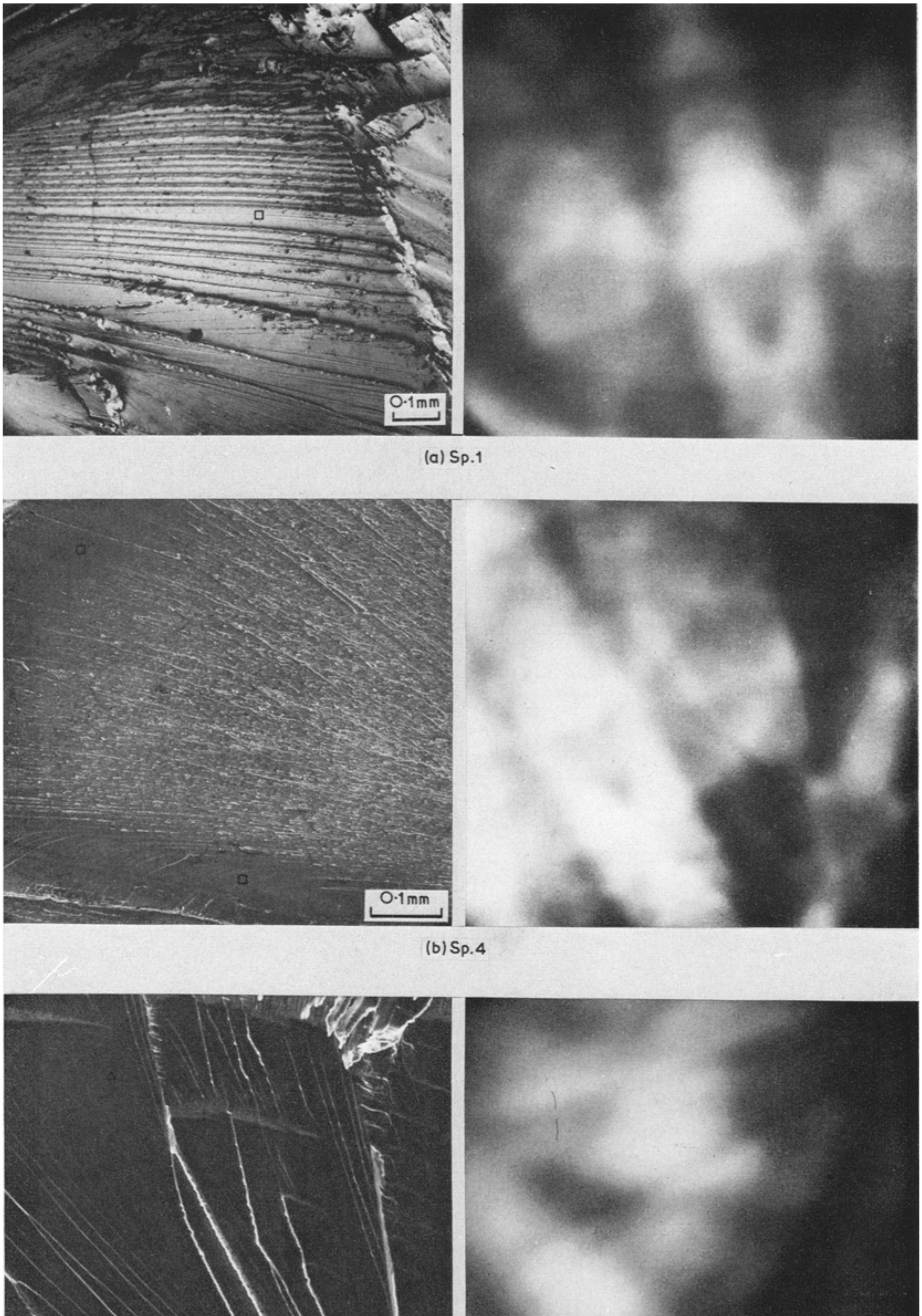
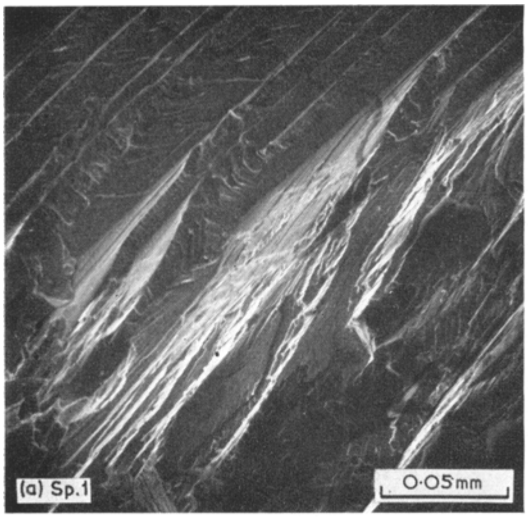
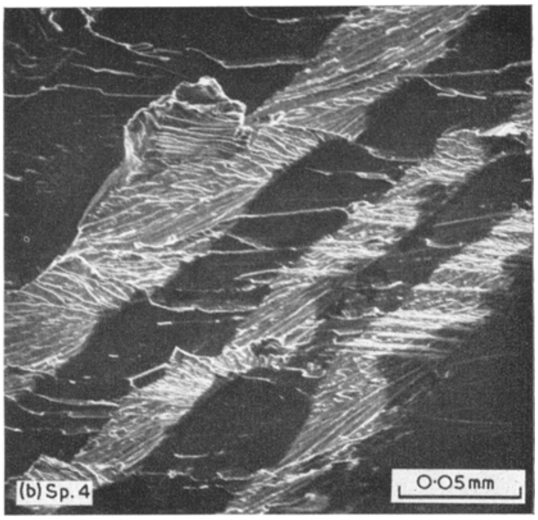


Figure 3 Some examples of the channelling patterns obtained from the fracture surfaces before annealing. Points where the patterns were made are shown as symbols (not to scale). Pattern resolution and contrast indicate ~ 3% equivalent strain present in specimens 1 and 4, and ~ 6% in specimen 6.





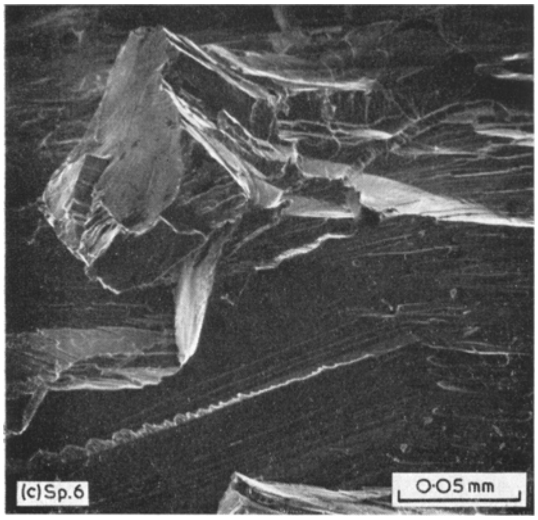


Figure 4 Fine cleavage features found on the same fracture surfaces as Fig. 2. These fine features indicate the same trends as found in Table I. The surface markings increase in size and density with increased plasticity. Note the plasticity associated with the crack in (c).

2 and 4 in order to obtain patterns from various fracture planes which could be positively identified (indicating strains of less than about 10%) whereas annealing was necessary to obtain positively identifiable patterns from the other specimens. Before annealing, however, some areas were found on all but specimens 7 and 12 which could be recognized. Another observation made in the course of taking channelling patterns from brittle looking cleavage facets is that strain within a facet is inhomogeneous; some areas indicate large deformation (> 10% strain), while other areas are nearly free of deformation. This condition does not appear to relate to fine surface topography details. Thus, it is apparently possible to get "cleavage" with varying amounts of accompanying plastic deformation. Some examples of cleavage observations, with associated channelling patterns (made from unannealed specimens), are shown in Fig. 3. Fig. 4 shows details of some of the cleavage facets observed on the same crystals, to illustrate how the fine features found on the surface reflect the macroscopic plasticity.

5. Summary and conclusions

Observations of fracture surface plasticity qualitatively agree with measured macroscopic plasticity prior to fracture, and electron channelling observations of fracture surface plasticity generally agree with the indications of plasticity found in topographic details. Macroscopic plasticity depends on the strain-rate and temperature of deformation, factors which also control plasticity at the tip of a propagating crack, along with stress state. But whereas macroscopic plasticity is relatively homogeneous throughout specimen gauge length, fracture surface plasticity is not; a single cleavage area may show wide variation in fracture surface plasticity, even though the surface topography was essentially the same over the whole area.

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