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I. INTRODUCTION

The possibility of substantial ductility in ionic solids was first demonstrated in 1958 for single crystals of a number of different materials having the sodium chloride structure.[1] Since that preliminary investigation, considerable work has been devoted to the development of ductile polycrystalline ceramic materials, and many papers have been published on the mechanical properties of ionic solids in both single and polycrystalline form.

However, the non-conducting properties of many of these materials, which give rise to their decomposition under prolonged electron bombardment, have resulted in only limited use being made of transmission electron microscopy. Following the initial work of Washburn et al.,[2,3] a number of papers have been published dealing with the electron microscopy of deformed MgO, but, almost without exception, these studies have been limited to single crystals. Recent work in this laboratory has demonstrated the feasibility of using transmission electron microscopy with polycrystalline MgO, provided the material approximates to theoretical density; the preliminary results of this research are outlined in this report.

II. MATERIALS AND PROCEDURE

Two types of material were used in this work, and these are shown in Fig. 1. The material on the left in this figure was produced in this laboratory by hot-pressing MgO powder with a 3% LiF additive, using the technique first developed by Rice.[4] Recent work by Benecke et al.[5] has indicated the conditions of temperature, pressure and time necessary during the hot-pressing to produce transparent specimens, approaching theoretical density, during the subsequent heat treatment. Specimens for the present investigation were prepared by hot-pressing for 3 hours at 1000°C under a constant pressure of 1200 psi, and then firing for 3 hours at 1300°C. This resulted in specimens having a measured Li content of approximately 75 ppm,* thereby showing that most of the LiF was in some manner removed during the densification process.[5] There was no apparent porosity in this material, as shown by the microstructure in Fig. 2(a), and the measured density, determined by displacement in water, was better than 99.5% theoretical on the basis of pure crystalline MgO.** The average grain size was of the order of 15μ. A typical polished specimen, measuring ∿0.25 x 0.25 x 1.0", is shown at the bottom of Fig. 1.

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* Analysis made by the American Spectroscopic Laboratories, San Francisco, California.

** Assuming a theoretical density for pure MgO of 3.5833 g/cc (Based on work by Skinner[6]).

The material on the right in this figure was obtained commercially (Honeywell Inc., Minneapolis, Minn.), and was produced by the isostatic pressing and sintering of high-purity MgO powder for 42 hours at a temperature of 1330°C. This resulted in an opaque material, having an average grain size of about 20u, and a density of the order of 98.5% theoretical. Many pores were visible in this material, as shown in Fig. 2(b), the majority of pores being located along the grain boundaries and at the triple points. It should be noted that the porosity is over-emphasized in the figure, owing to the tendency for the pores to open up during the polishing procedure.

To prepare thin foils for the electron microscope, specimens were cut on the diamond saw to pieces of ∿0.012" thickness, and then thinned chemically in ortho-phosphoric acid at 150°C. Since MgO is transparent to light, thin portions suitable for use in the electron microscope (∿1000 Å thickness) were selected by examining the specimens in the optical microscope after holes had first appeared. The very thin portions were then visible at the edges of the holes by the occurrence of interference fringes. A major problem encountered with this process was that the commercial material, having the high porosity, was extremely difficult to thin satisfactorily, since preferential attack occurred to open up the pores. As a result, whole grains were often removed from the edge of the foil, and it was not easy to use this material for examination of the boundary regions. The major portion of the electron microscopy has therefore been concentrated on examining foils of the high-density hot-pressed material. All specimens were examined in either a Hitachi HU-10 or a Siemens Elmiskop I electron microscope, operating at 100 kv.

Further details concerning the preparation of thin foils of polycrystalline MgO are given elsewhere.[7]

III. RESULTS

A primary objective of this work was to investigate the possibility of the LiF additive forming a thin layer of some second phase along the grain boundaries of the hot-pressed material. No such phase was detectable using optical microscopy, and a close examination of the grain boundaries in the unstrained material using electron microscopy failed to reveal the presence of any such phase under this higher magnification. A typical grain boundary in the hot-pressed material is shown in Fig. 3, in which the boundary is at an angle with the plane of the foil.

In general, the grains were relatively dislocation-free, as shown in Fig. 4, although there were a few isolated grown-in dislocations in some grains. Care was taken not to confuse the grown-in dislocations with any fresh dislocations put into the foils during their preparation. In Fig. 4, for example, dislocations have been generated from the grain boundary at point A, but this is almost certainly due to the accidental cutting of the foil along line AB whilst separating it from the parent material.

The presence of pores in the sintered material made the thinning process difficult, but some suitable thin foils were obtained. A typical example is shown in Fig. 5, in which small pores are visible in the boundary at A. As with the hot-pressed material, there was no evidence of a second phase along the boundaries in the small number of foils examined.

To study the deformation mechanisms in polycrystalline MgO, specimens were tested under compression in air at temperatures in the range from room temperature to 1400°C. Specimens were loaded under conditions of constant force rate, with an initial rate of 20 psi/sec., and the strain was continuously recorded by measurements taken directly on the specimen. Full details of the equipment, and method of testing, are given elsewhere.[8] All specimens were tested after careful chemical polishing, since it is known that submicroscopic damage can markedly affect the deformation characteristics of this material.[9]

The resultant stress-strain curves showed that at temperatures less than about 1200°C, for both the hot-pressed and sintered material, the ductility was limited, and specimens fractured at total strains of less than ∿0.5%. At temperatures of 1200°C and above, a sharp yield point was observed at total stresses in the range 10,000-25,000 psi, followed by a very rapid rate of strain. At the highest temperatures, specimens ultimately fractured at total strains in excess of 10%. In compression at temperatures less than ∿1200°C, slip is confined to the $\{110\}\ \langle 1\bar{1}0\rangle$ slip systems, giving rise to straight slip traces on the specimen surface. At temperatures about ∿1200°C, however, slip can also occur on the $\{001\}\ \langle 1\bar{1}0\rangle$ systems, and 90° intersections of the $\{110\}\ \langle 1\bar{1}0\rangle$ slip systems become easy, so that wavy slip traces are observed.

A similar transition to multiple slip has also been reported for polycrystalline MgO tested in tension, but the temperature at which this occurs is then 1700°C for recrystallized MgO and 2200°C for hot-pressed material.[10] In both tension and compression, the situation is further

complicated by the existence of grain boundary sliding at the higher temperatures, which may lead ultimately to intergranular failure. Direct evidence of sliding, and intergranular cavitation, has already been presented for polycrystalline MgO tested in compression.[11,12] Furthermore, recent work by Mountvala and Murray[13] has shown that a sharp drop in grain boundary fracture stress exists at about 1200°C for twist boundaries of MgO bicrystals tested in compression. In this work, grain boundary sliding was inferred from the lack of transparency in the hot-pressed specimens after testing at the higher temperatures.

At temperatures less than 1200°C, specimens were strained to ∿0.1%, and then examined in the electron microscope prior to fracture. Relatively few dislocations were observed in these specimens after deformation, thereby confirming that only a limited amount of slip preceded fracture. This behavior is a direct result of the limited slip systems then operative, which provide for only two independent systems. At temperatures of 1200°C and above, five independent slip systems exist potentially, and, by the von Misés criterion,[14] the specimens should then be able to deform homogeneously. However, the problem of intersection of the slip systems still exists. A large number of dislocations were visible in specimens deformed at these temperatures, and, since bulk diffusion is then possible, the dislocations were able to climb out of their slip planes to annihilate or polygonize. The typical dislocation structure for a specimen deformed ∿1.5% at 1400°C is shown in Fig. 6. A small angle boundary is visible in this figure at A.

IV. CONCLUSIONS

The preliminary results of this investigation show clearly that transmission electron microscopy is a useful tool for studying the deformation mechanisms in polycrystalline MgO. However, it is imperative that the specimens used approximate to theoretical density, owing to the difficulty of preparing thin foils of even slightly porous material.

ACKNOWLEDGMENTS

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FIGURE CAPTIONS

Fig. 1. Polycrystalline MgO produced in this laboratory by hot-pressing (left) and produced commercially by sintering (right).

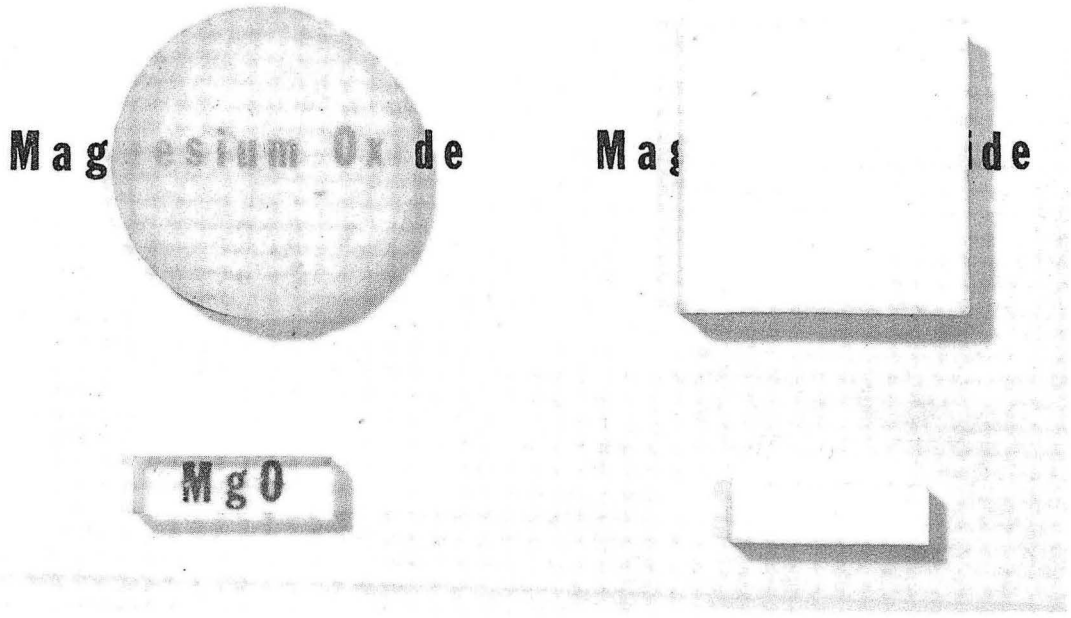
Fig. 2. Microstructure of (a) hot-pressed material and (b) sintered material.

Fig. 3. Typical grain boundary in unstrained hot-pressed material; the boundary is at an angle with the plane of the foil.

Fig. 4. Relatively dislocation-free grains in unstrained hot-pressed material. Dislocations may be generated by cutting of the foil, as at A.

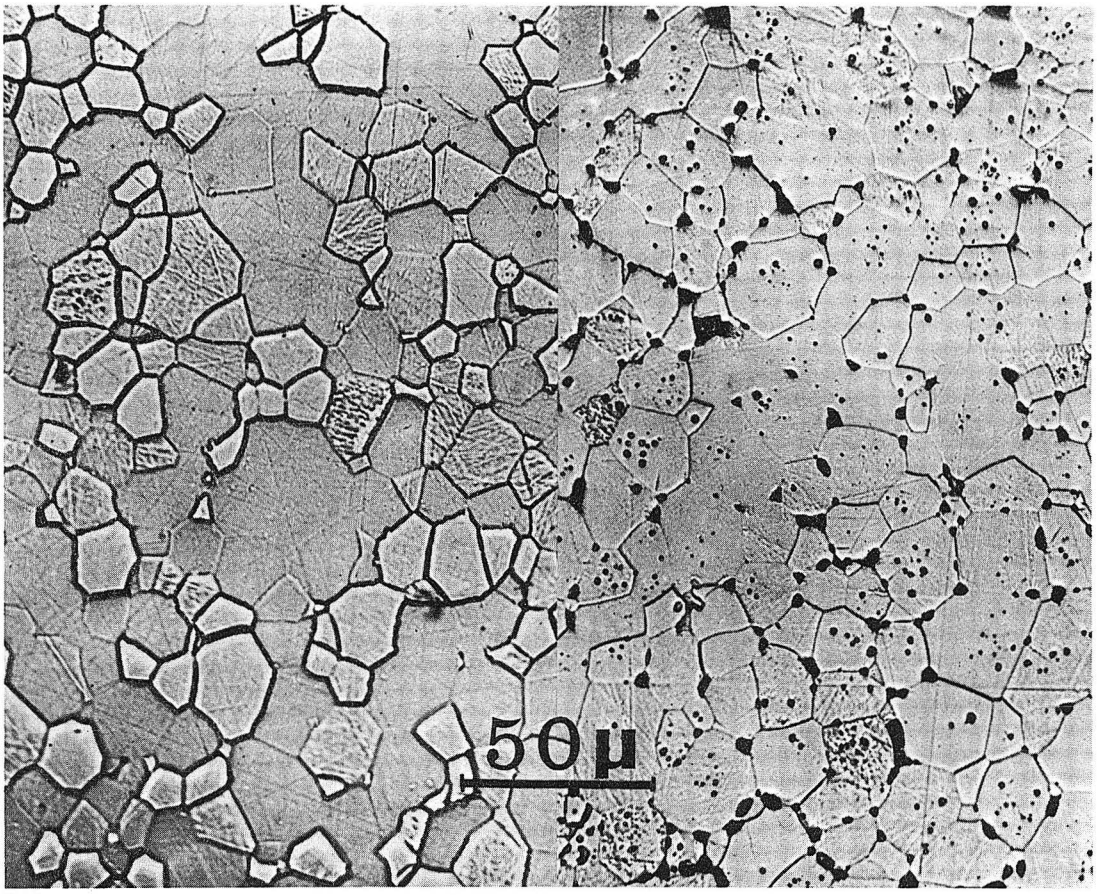
Fig. 5. Triple point in unstrained sintered material. Pores are visible in the boundary at A.

Fig. 6. Dislocation structure in hot-pressed material deformed ∿1.5% at 1400°C. A small angle boundary is visible at A.



ZN-6010

Fig. 1



ZN-6011

Fig. 2

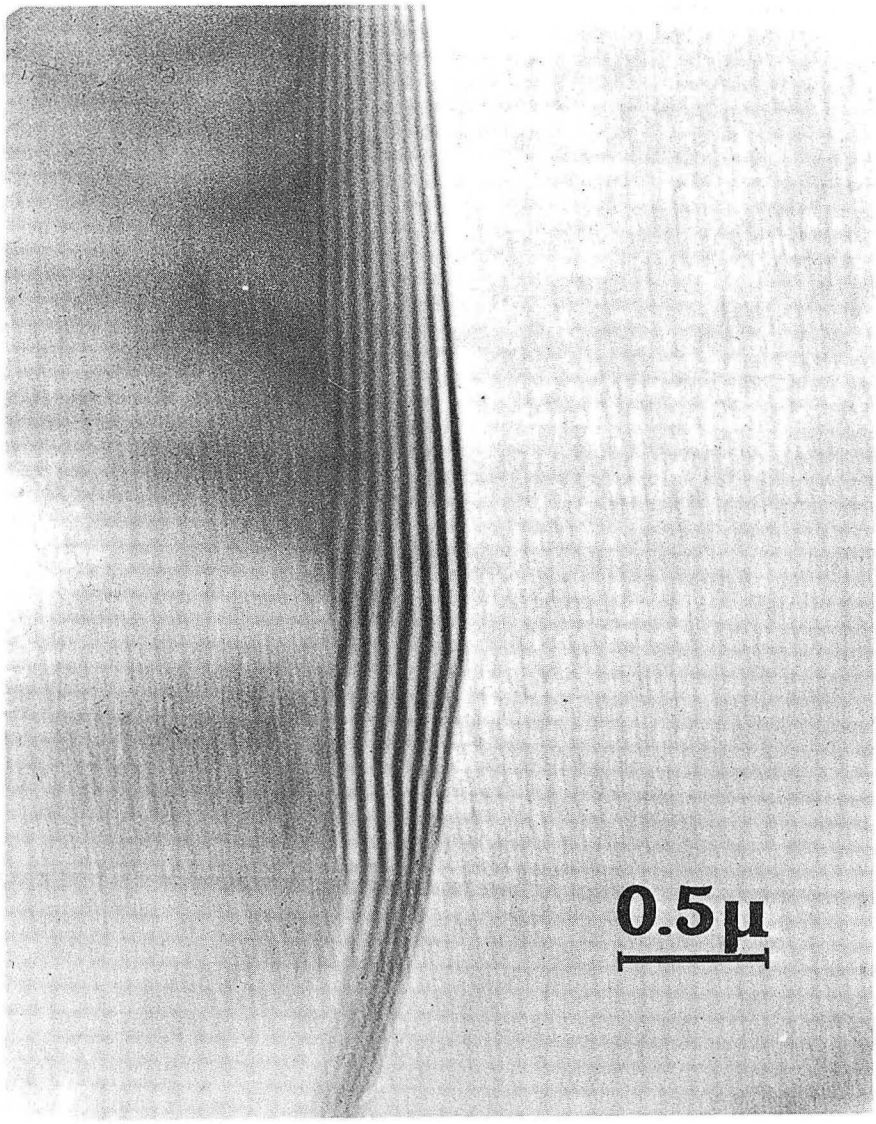


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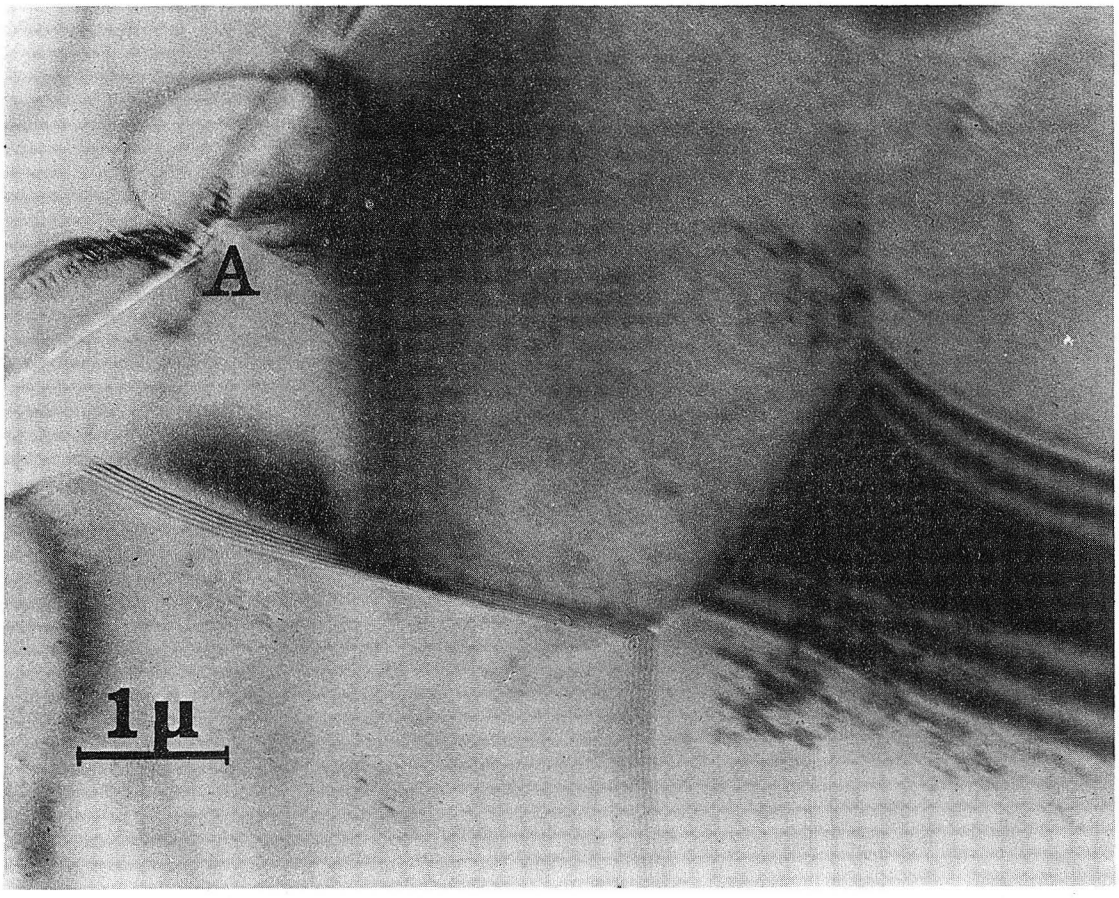
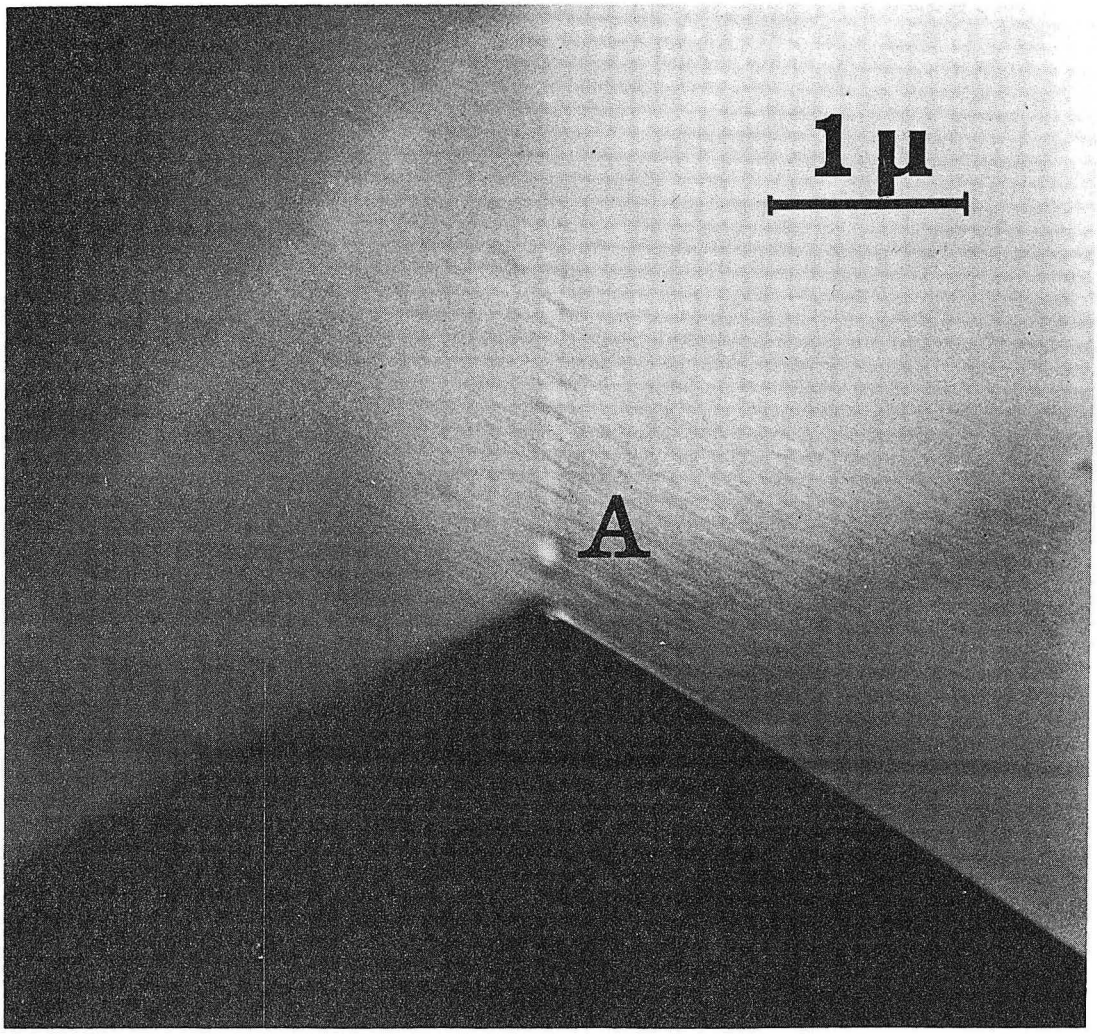
Fig. 3



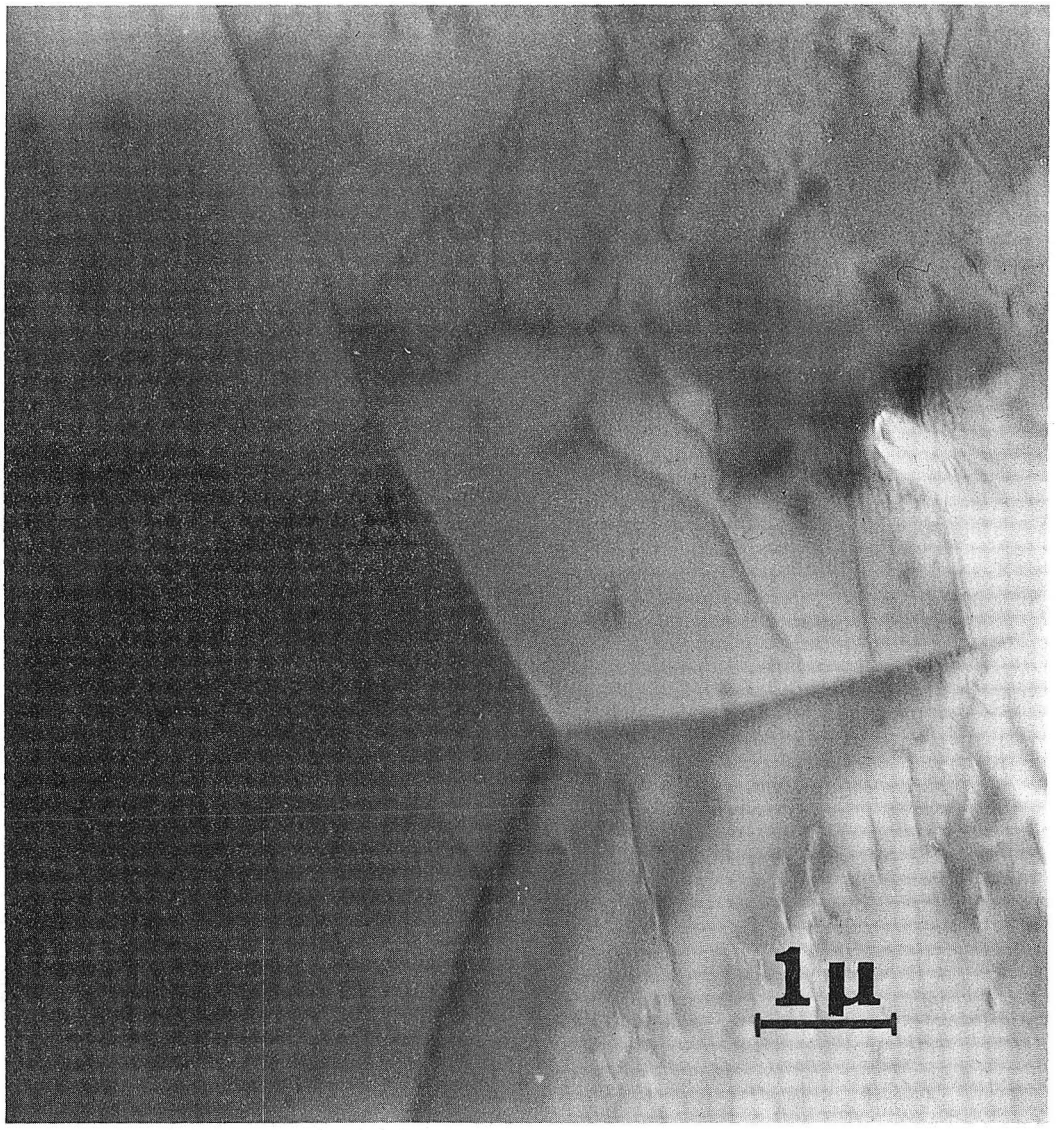
ZN-6013

Fig. 4



ZN-6014

Fig. 5



ZN-6015

Fig. 6

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