Figure 5 Flow curves of annealed Cu/40% Zn alloy. Note psi units are used in this diagram. 1.0 psi = 1.0 lb/in.^{2} = 7.0 × 10^{-2} kg/cm^2.

haviour, whether from the crystal structure or the metallurgical condition, is sufficient to cause the breakdown of Tabor's correlation.

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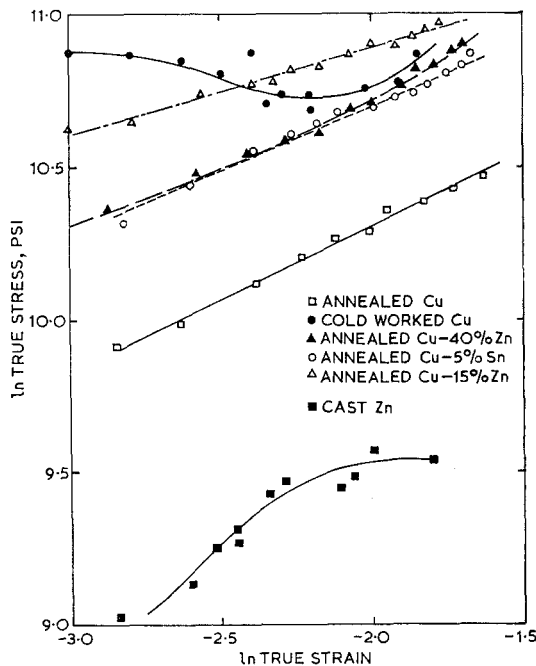   *Eastlands, Rugby, UK*

Figure 6 Logarithmic form of flow curves derived from hardness measurements. Note psi units are used in this diagram. 1.0 psi = 1.0 lb/in.^{2} = 7.0 × 10^{-2} kg/cm^2.

Visually Observed Pressure-Induced Transformation Behaviour

The visual observation of temperature-induced phase transformations has been of great assistance in the characterisation of their mechanisms. Similar observations on pressure-induced transformations are limited [1], and in fact, the transformation mechanisms of pressure-induced changes are presently rather obscure. In the present work, a diamond high-pressure cell was used to obtain qualitative descriptions of pressure-induced phase changes in transparent materials; the study indicates that, as in the case of temperature-induced phenomena, both nucleation-and-growth and martensitic-type transformations can occur.

The diamond cell and its utility has been described in detail in the literature [1-7]. Essentially, two diamonds serve as anvils; the smaller diamond has a surface area of approximately 0.16 mm^2. Loading is provided by a hand-operated turn-screw. Desired radiation passes through the diamonds parallel to the

uniaxial loading. Appropriately gasketed liquids, prepared by previously described techniques [8], were the starting samples. Observations were made using a transmitted-light, polarising microscope at a magnification of about 200×. Characteristic behaviour provided by the transformations in n-butanol [9], carbon tetrachloride [10], and benzene [11] are described.

Pressure-induced solidification occurred in all materials by a nucleation-and-growth process, resulting in the formation of a polycrystalline solid. The growth stage for carbon tetrachloride was so rapid that in many tests the transformation took place unnoticed. In these instances the occurrence of solidification could only be confirmed by the observation of melting upon pressure reduction. The observation of the solidification of benzene was greatly aided by the anisotropy of the resulting solid, producing strong interference colours when viewed under crossed Nicols. In contrast to carbon tetrachloride and benzene, whose initial solid phases had equiaxed structures, solidification of butanol occurred by heterogeneous nucleation at the gasket-liquid interface followed by slow needle-like growth.

Carbon tetrachloride [10] undergoes two pressure-induced solid state transformations,

I-II at about 4 kbar and II-III at about 7 kbar. (Bridgman's designations for solid phases will be utilised throughout.) The I-II transformation is generally characterised by heterogeneous nucleation at the gasket-sample interface, followed by slow interface growth. This interface and resultant phase II are shown in fig. 1a, wherein the direction of growth is indicated. Note that the shape of the interface, specifically the three regions of curvature, shown in fig. 1a, is compatible with the three-grain structure of phase II as seen under crossed Nicols in fig. 1b. Again, birefringence is extremely useful in making the transformation and the resultant grain structure more recognisable since the three-grain structure is not identifiable without crossed Nicols (see fig. 1a). The growth rate of phase II was found to be highly dependent on departures from equilibrium pressure so that when a high degree of metastability existed, growth was quite rapid. Nucleation of phase III occurs at grain boundaries and is followed by slow growth.

The solid state transformation in benzene was strikingly different in its basic growth mechanism from that in carbon tetrachloride. Seemingly instantaneous growth of intragranular lenticular platelets was observed. The initial stage is shown in fig. 2a; at constant
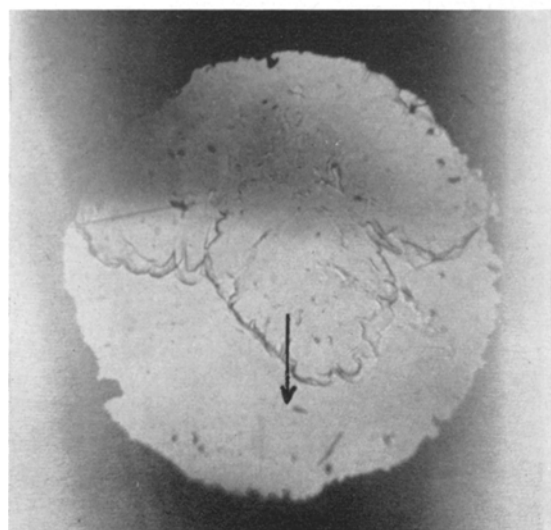
(a)   (b)

Figure 1 Solid-solid transformation in carbon tetrachloride (×200). (a) During I-II at 3.6 kbar (arrow indicative of growth direction). (b) Resultant phase II (crossed Nicols).

pressure, no additional growth was observed during 60 h. Upon subsequent pressure increases, striations suddenly appear in other regions of the microstructure; the resulting appearance is shown in fig. 2b. The athermal kinetics, the geometry of phase II, and the apparent crystallographic relationship between phases I and II suggest that this is a displacive transformation, commonly designated as martensitic.

Interestingly, the characteristics of the reverse transformation in benzene during pressure unloading are quite different. At constant pressure, the striations rapidly disappear and regions form which begin to grow slowly (figs. 2c and 2d). Upon further pressure reduction, the interface regions between newly formed grains become highly mobile and grow rapidly to a resultant grain structure which differs from that indicated in fig. 2a. These changes suggest that a nucleation and growth mechanism is occurring,
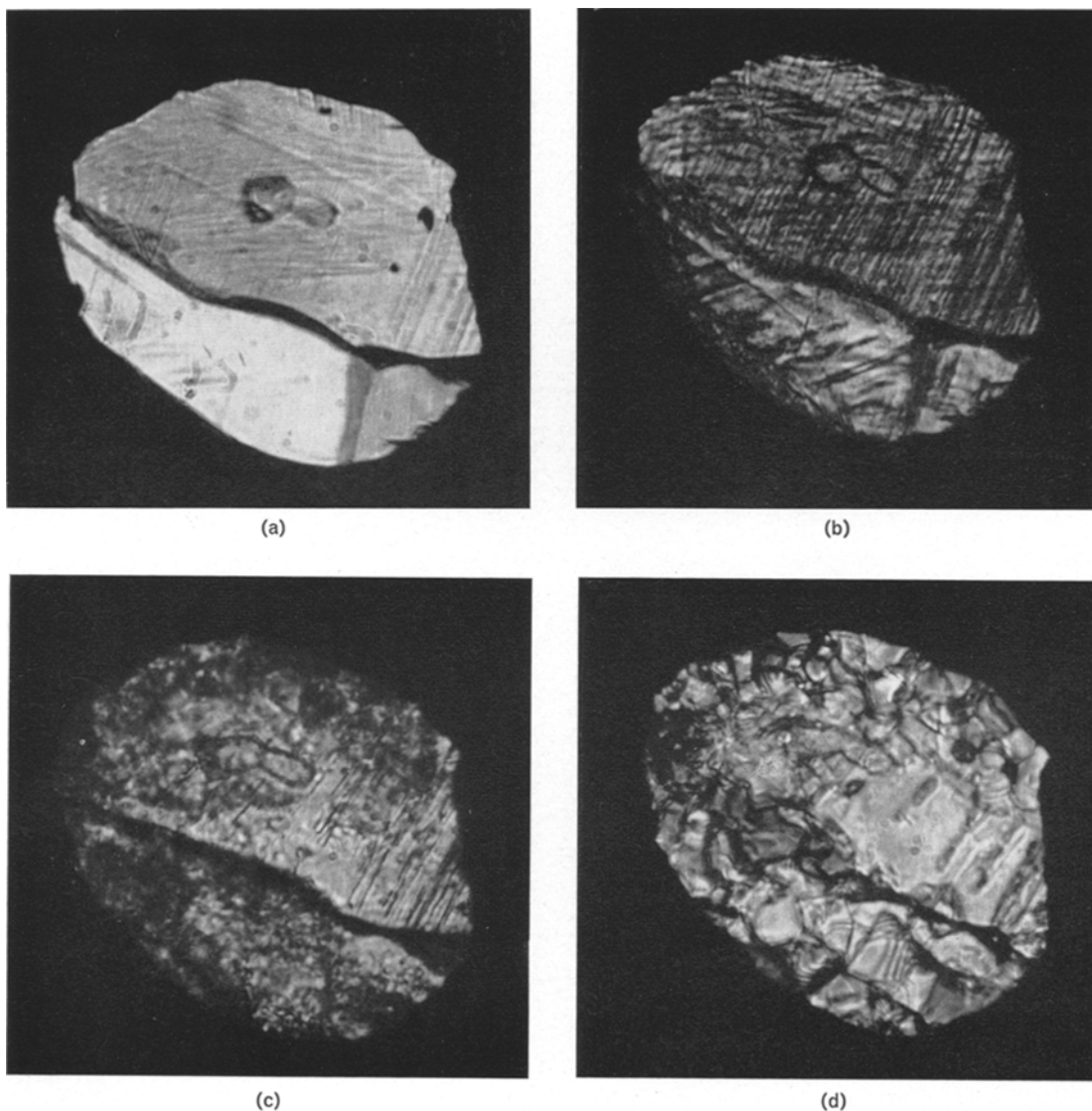

(a)

(b)

(c)

(d)

Figure 2 Solid-solid transformation in benzene (×250, crossed Nicols). (a) Initial stages of I-II at 12 kbar. (b) Resultant phase II after additional pressure application. (c) Initial change during pressure reduction. (d) Further change after 3 min at same pressure as 2c.

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although its complexity indicates that visual observation alone is insufficient for definitive characterisation. Differences in the stress system imposed during essentially uniaxial loading as compared to that existing during unloading may account for the observed differences between loading and unloading transformations. The difference between loading and unloading stress systems would be less significant for nucleation and growth transformations; thus for carbon tetrachloride both loading and unloading characteristics were the same.

Acknowledgements

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On the Formation of Grain-Boundary Cracks during the Deformation of Zinc Bicrystals at Elevated Temperatures

Zinc bicrystals with a high-angle tilt grain-boundary of $2\theta = 84°$ (fig. 1) and containing a $[11\bar{2}0]$ slip direction common to both crystals
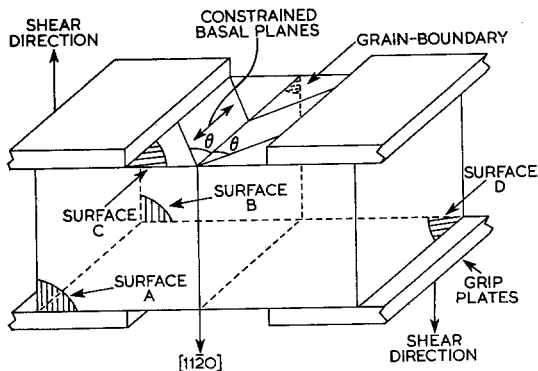

Figure 1 The bicrystal orientation, nomenclature and loading configuration.

in the plane of the boundary, were grown from the melt in two grades of zinc containing 100

ppm and 1 ppm impurities (spectrographic analyses are presented in table I). Specimens of approximate dimensions $2 \times 0.65 \times 0.25$ cm were obtained from the as-grown bicrystals by chemical sawing and electropolishing to ensure deformation-free surfaces. The specimens were loaded in shear in the sense shown in fig. 1, using a gripping arrangement which resulted in slip on basal planes intersecting both the grain-boundary and the grips. Tests were performed in an atmosphere of high-purity argon over the temperature range 20 to 175° C at a constant rate of grip displacement of 300 μm/h.

TABLE I Spectrographic analyses of zinc bicrystals (in ppm).

	Ag	Cd	Cu	Fe	Pb
4-9's zinc	1	5	10	5	10
6-9's zinc	—	0.1	0.1	0.1	0.1

The deformation behaviour of the bicrystals over the temperature range 20 to 175° C is presented in fig. 2 in terms of the shear stress on the grain-boundary versus grip displacement. The discontinuous changes in the curves are attributable to the formation of kink bands