HYDROSTATIC PRESSURE INDUCED DUCTILITY TRANSITIONS IN PURE BISMUTH AND TIN-BISMUTH ALLOYS*

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The mechanical behavior of pure (99.999%) bismuth and tin-bismuth alloys of various compositions has been observed over a range of superimposed hydrostatic pressures. Results indicate that maxima in ductility (as measured by percent reduction in each at the fracture surface) in specimens tested at atmospheric pressure occur at compositions bordering pure tin and the eutectic compositions. At sufficiently high pressures all compositions failed by rupture, i.e. necking to virtually 100 per cent RA. For pure bismuth, pressure was observed to retard failure due to the formation of cracks at twin-grain boundary interactions; this result was consistent with the hypothesis that the effect of pressure is to shift the mode of crack propagation by decreasing the normal tensile component of stress acting on a crack.

TRANSITIONS DE DUCTILITE DUES A UNE PRESSION HYDROSTATIQUE DANS LE BISMUTH PUR ET DANS LES ALLIAGES ETAIN-BISMUTH

Les auteurs ont étudié les propriétés mécaniques du bismuth pur (99,999%) et de divers alliages étainbismuth en présence de pressions hydrostatiques. Les résultats indiquent que pour les échantillons déformés à la pression atmosphérique, la ductilité maximale (mesurée par la réduction de section en % à la surface de rupture) se produit pour des compositions voisines de l'étain pur et des eutectiques. Pour des pressions suffisantes, tous les alliages cassent avec des strictions de pratiquement 100%. Dans le bismuth pur, la pression retarde la cassure due à la formation de fissures aux intersections de joints de grains et de joints de macle; ce résultat s'accorde avec l'hypothèse que la pression change le mode de propagation d'une fissure en diminuant la contrainte normale agissant sur elle.

DUKTILITÄTS-ÜBERGÄNGE IN REINEM WISMUT UND IN ZINN-WISMUT-LEGIERUNGEN UNTER EINEM HYDROSTATISCHEN DRUCK

Das mechanische Verhalten von reinem Wismut (99,999%) und von Zinn-Wismut-Legierungen wurde bei verschiedenen hydrostatischen Drucken beobachtet. Die Ergebnisse zeigen, daß Duktilitätsmaxima (Meßgröße ist prozentuale Reduzierung an Bruchfläche) in den bei Atmosphärendruck untersuchten Proben in nahezu reinem Zinn und in Proben mit nahezu eutektischer Zusammensetzung auftreten. Bei genügend hohen Drucken kamen Proben aller Zusammensetzungen zum Bruch, d.h. Halsbildung bis virtuell 100% RA. In reinem Wismut verzögert hydrostatischer Druck den Bruch aufgrund einer Rißbildung an Zwilling-Korngrenzen-Schnittlinien. Dieses Ergebnis ist in Übereinstimmung mit der Hypothese, daß der Druck den Rißausbreitungsmechanismus durch ein Verminderung der normalen auf den Riß wirkenden Zugspannungskomponente verschiebt.

INTRODUCTION

In general, it has been observed that ductility, measured by either percent reduction in area at the fracture surface or the natural strain to fracture $(\ln{(A_0/A_f)})$, is increased by a superimposed hydrostatic pressure. The ductility pressure function can be either linear with a characteristic slope,^(1,2) nonlinear,^(3,4) or abruptly increasing within a limited region of pressure.^(5–7) A comprehensive review of the effects of pressure on mechanical properties of materials and techniques for determining these effects will not be attempted here, and the reader is referred to a review by Brandes⁽⁸⁾ for this information.

Most investigations concerning the effects of pressure on the ductility of materials have been limited to single phase alloys or pure materials. In this current study, a two phase alloy, wherin the two phases exhibit marked differences in intrinsic ductility and pressure response is examined. Chosen for study was the tin-bismuth alloy system. Here the nature of the system is such that there is little room temperature solid solubility and the existence of a eutectic reaction permits variation of mixture morphology.

Prior work⁽⁹⁾ has shown that bismuth undergoes an abrupt increase in ductility over a narrow pressure range. It has also been reported⁽¹⁰⁾ that a 50 at.% Sn–50 at.% Bi alloy shows a similar abrupt transition, but that further increases in pressure above the transition pressure results in a decrease in ductility. Such a phenomena is contrary to the observed behavior of many other materials and inconsistent with proposed explanations of the pressure effect on ductility.

Reported herein is the response of ductility to pressure for several Sn–Bi compositions and microstructures. The pressure effects upon ductility are interpreted in terms of the fracture modes observed.

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TABLE 1. Fabrication conditions

Composition	Method extrusion	Temperature	Post extrusion treatment	Lubricant
100% Bi	Hot	150°C	a. Air cooled b. Water quenched	None
9% Sn-bal Bi	Hot	150°C	Air Cooled	None
30% Sn-bal Bi	Hydrostatic	Ambient	None	Pb-MoS ₂
42% Sn-bal Bi	Hydrostatic	Ambient	None	None
75% Sn-bal Bi	Hydrostatic	Ambient	None	Pb-MoS ₂
90% Sn-bal Bi	Hydrostatic	Ambient	None	None

Note: Virtually no change in intercept grain size as a function of cooling rate was observed in pure bismuth.

MATERIALS AND PROCEDURE

The bismuth and tin used in the fabrication of the alloy compositions were reagent grade stock; the nominal purity level of the former being 99.8 per cent Bi and the latter 99.9 per cent Sn. The material used for investigating the pressure–ductility response of pure bismuth was of high purity (99.999 per cent) $\frac{1}{2}$ in. polycrystalline bar stock. Alloys were prepared by melting in a Pyrex crucible open to the atmosphere and using SnCl_2 as a flux. Wherever possible, bismuth was added to the tin to minimize oxidation. Fabrication conditions for bar stock are given in Table 1 and the specimen annealing schedule in Table 2. All temperatures listed were held to within $\pm 2^{\circ}\mathrm{C}$. All specimens were packed in fire clay powder prior to annealing.

Specimens were cylindrical in shape, having a nominal gauge length of 0.562 in. (the shoulder to shoulder distance) and diameters of either 0.160 (pure bismuth) or 0.090 in. (tin-bismuth alloys). The apparatus used in the course of the investigation was a 30 kb Bridgman-Birch hydrostatic system which has been previously described; (7) pressure measurement was accomplished through the use of a manganin wire transducer used in conjunction with a Foxboro recorder. The estimated error in pressure measurement was ± 2 per cent.

Extension of the specimen was accomplished

through use of the fixture shown in Fig. 1. Tensile force is introduced into the specimen by the advance of the main piston into the pressure cavity, with this motion in turn transmitted into the specimen by two movable legs. The other two legs are stationary and bear against the bottom closure of the high pressure cavity, thereby fixing the position of one end of the specimen. Since straining of the specimen is accomplished by the advance of the piston into the high pressure cavity, the tests are not isobaric and an increase in pressure occurs as the specimen is extended. In reporting data the pressure at fracture is plotted. Crosshead movement was estimated from a record of piston displacement and was maintained at a rate of 0.05 in/min. All tests were performed at ambient temperatures.

Due to the relative softness of the materials used, mounting specimens for metallographic examination in an epoxy compound (Hysol) was adequate to insure reasonably good edge retention. Pure bismuth was examined in the as polished condition using polarized light and the Sn–Bi alloys were examined in either the as polished condition, or were etched using a solution of 10 ml hydrochloric acid and 0.5 g chromic acid in 250 ml of water prior to viewing. Electron micrographs were prepared using a two stage replication technique employing chromium as a shadowing material.

Table 2. Annealing schedule for Bi and Sn-Bi alloys

Composition	Condition prior to annealing	Annealing schedule	Comments
100% Bi	Hot extruded and air cooled Hot extruded and water quenched	None None	If the extrusion temp. is 66C, the grain dia immediately after extrusion is very small and increases to approximately 12.5 mm in 24 hr
9% Sn-bal Bi 30% Sn-bal Bi	Hot extruded and air cooled Hydrostatically extruded	ST (a) None	
30 /0 BH-bar Di	lly drostationly extraded	(b) ST	Pb-MoS, coating not removed
42% Sn-bal Bi	do.	132C for 25 hr, air cooled	
75% Sn-bal Bi	do.	\mathbf{ST}	
90% Sn-bal Bi	do.	ST	The state of the s

Note: "ST" is defined as heating at 120C for 235 hr, quenching into water at 0°C, upquenching to 24C for 14 hr and a second upquench to 66C for 4 hr, followed by air cooling to room temperature.