Effect of relatively hard particles on caviation of microduplex Pb–Sn eutectic during superplastic flow

During recent studies on microduplex alloy steels containing titanium, cavitation was observed during superplastic tensile deformation [1–3]. Cavity growth and coalescence resulted in premature failure, even though substantial elongations could be achieved. Metallographic and density studies led to the view that Ti(C, N) particles were playing a significant role in cavity nucleation [2, 3].

To identify more clearly the role played by hard particles on cavity nucleation during superplastic flow it was decided to study the effect of such particles on the behaviour of an alloy which did not normally undergo cavitation. Lead–tin eutectic appeared to provide the basis of an ideal model system in that it could be readily obtained in a microduplex form, it was superplastic at room temperature and was capable of giving high tensile elongations, and it did not appear to undergo cavitation [4]. Particles were introduced in the form of the intermetallic phase believed to be Ag_3Sn, by making silver additions to the eutectic alloy.

An Sn–38.1 wt% Pb eutectic alloy and an Sn–36 wt% Pb–5.6 wt% Ag alloy were prepared by induction melting the components (> 99.99% purity) under argon in a graphite crucible. The alloys were chill cast into 13 mm bars, flat rolled at room temperature to 1 mm thick sheet, and tensile test pieces of 10 mm gauge length and 5 mm width were stamped out. The test pieces were annealed at 100° C for 1 h to develop the required microstructure, and then stored at 0° C to prevent grain growth prior to testing [4].

Metallorgraphic examination showed the Sn–Pb eutectic had a microduplex structure with an average grain size of ~ 2 μm while the ternary alloy had a similar matrix structure but also contained ~ 5% by volume of particles of rectangular or rounded shape with average dimensions of 3 μm. Microprobe analysis confirmed that these particles were silver-rich, and they were assumed to be based on Ag_3Sn. Vickers microhardness results obtained from a coarsened microstructure gave the following results: Ag_3Sn 77 VPN, Sn-rich phase 3 VPN, Pb-rich phase 2 VPN.

The relationships between flow stress and strain-rate for the two alloys, which were obtained by successively changing the cross-head velocity of an Instron tensile machine and recording the flow load at each velocity, are plotted logarithmically in Fig. 1. The slopes of the curves give values of the strain-rate sensitivity, m, and it was observed that the silver addition reduced the maximum value of m from 0.53 to 0.40, and also increased the stress level for a given strain-rate. However, the value of m for the ternary alloy is consistent with superplastic behaviour.

Test pieces of the two alloys were tested to failure at various constant cross-head velocities. The results of the tests are given in Table I while the silhouettes of the fractured specimens are shown in Figs. 2 and 3. It can be seen for the three velocities examined that Sn–Pb shows substantially greater elongations than the Sn–Pb–Ag alloy. A

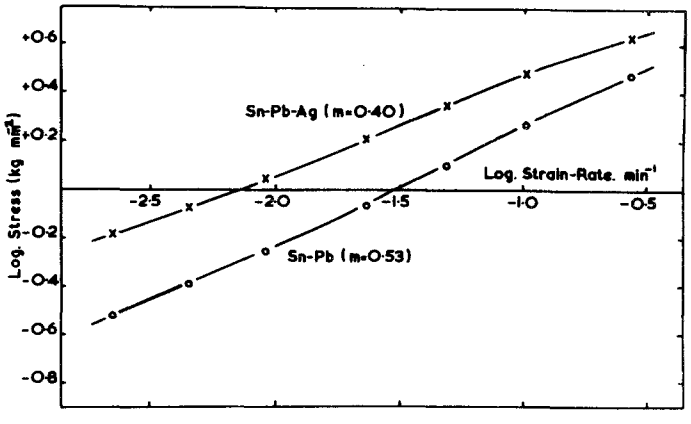

Figure 1 Logarithmic plot of flow stress versus strain rate for Sn–Pb and Sn–Pb–Ag alloys.

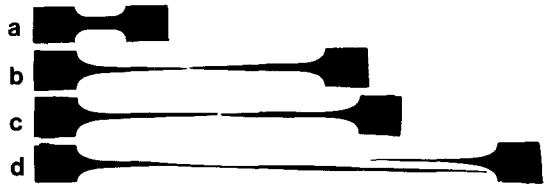

Figure 2 Silhouettesof Sn–Pb tensile specimens tested to failure at various constant cross-head velocities: (a) undeformed, (b) 0.2 cm min^{-1}, (c) 0.1 cm min^{-1}, (d) 0.05 cm min^{-1}.

Figure 3 Silhouettes of Sn–Pb–Ag tensile specimens tested to failure at various constant cross-head velocities (a) undeformed, (b) 0.2 cm min^{-1}, (c) 0.1 cm min^{-1}, (d) 0.05 cm min^{-1}.

TABLE I Elongations to fracture of Pb–Sn and Pb–Sn–Ag specimens at various constant cross-head velocities

Constant cross-head velocity (cm min^{-1})	Elongation % to fracture	
	Sn–Pb	Sn–Pb–Ag
0.2	806	250
0.1	854	344
0.05	1812	362

further significant feature is shown in Fig. 2 where it can be seen that the broken Sn–Pb specimens have necked down to a fine point or to a relatively fine point, whereas the fracture faces of the ternary alloy have a substantial cross-sectional area. This latter feature is consistent with a premature brittle-type failure due to the nucleation, growth and interlinkage of cavities [5].

Specimens for density measurement were strained at a cross-head velocity of 0.1 cm min^{-1} to elongations of 100, 200 and 300%. The essentially parallel-sided gauge lengths, and the gauge heads, were cut from the deformed specimens. After electropolishing the densities of the gauge lengths were measured relative to those of the undeformed heads by hydrostatic weighing in ethyl iodide maintained at constant temperature, and the differences expressed as % void volume. For the Pb–Sn alloys there was no significant difference in density between the heads and gauge length ($< 0.05\%$) whereas cavitation had clearly occurred in the alloys containing silver (Table II).

TABLE II Volume of voids in Pb–Sn and Pb–Sn–Ag at various elongations

Elongation (%)	Volume of voids (%)	
	Sn–Pb	Sn–Pb–Ag
100	< 0.05	0.08
200	< 0.05	0.96
300	< 0.05	1.40

The absence of cavities in the Sn–Pb alloys and their presence in the Sn–Pb–Ag alloys was confirmed by metallographic examination of superplastically deformed specimens. The Sn–Pb alloys remained equiaxed, although grain growth had occurred during deformation. In the Sn–Pb–Ag alloys it could be seen that cavitation had occurred at many of the Ag_3Sn–matrix interfaces (Fig. 4), a situation which was analogous to that in steels containing Ti(C, N) particles. The only structural difference between the Sn–Pb–Ag alloy and the steel was that the former contained ~5% volume of hard particles, whereas in the steels the volume would rarely exceed 1%.

Nevertheless, it has been clearly illustrated that during superplastic tensile deformation, cavitation, leading to premature failure, can be induced in systems that do not normally cavitate, if relatively hard particles are introduced.

Acknowledgement

C.W.H. is grateful to the British Steel Corporation (Physical Metallurgy Centre) for financial support.

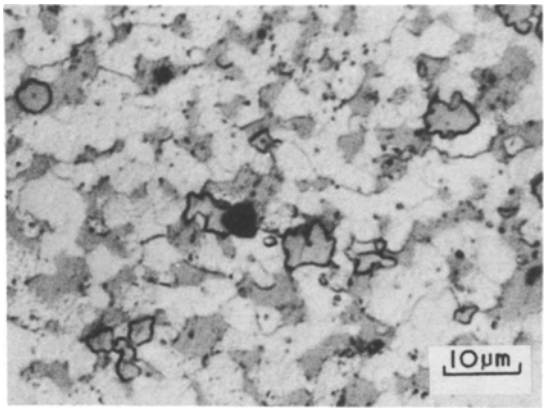

Figure 4 Cavitation at Ag_3Sn–matrix interfaces in the Pb–Sn–Ag alloy.

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Received 29 October
and accepted 1 December 1976

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The magnetic properties of Mn_2Sb modified with vanadium

Swoboda *et al.* [1] found that the addition of chromium can induce Mn_2Sb to show, on cooling, an abrupt transition from its normal ferrimagnetic state [2] to an antiferromagnetic one (the type of behaviour shown in Fig. 1). This phenomenon is found in a limited number of other materials [3]. Bither *et al.* [4] later reported further elements which modify the magnetic properties of Mn_2Sb in the same way. Vanadium additions [4] seem particularly interesting because, in the region of ambient temperature, the magnetic transition is relatively insensitive to composition (Fig. 2). The present work was designed to supplement the data given by Bither *et al.*, particularly at higher vanadium levels.

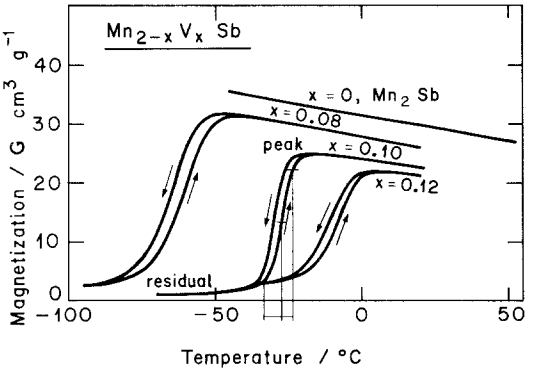

Figure 1 The magnetic transition in some vanadium-modified Mn_2Sb alloys on heating and cooling. The range of temperature used in Fig. 2 to describe the transition on heating, corresponding to 0.1, 0.5 and 0.9 of the magnetization between the residual and peak values, is also indicated.

The alloys studied here were made by melting the components together under argon in a pyrolytic boron nitride crucible using intermediate frequency heating. The compositions $Mn_{2-x}V_xSb$ were in the range $x = 0.08$ to 0.25. The alloys were annealed in vacuum at 700° C for 2 weeks. Polycrystalline spheres (~ 2 mm diameter) were subsequently ground out and tested in a vibrating sample magnetometer with a heating rate of 5° C min^{-1}. The applied field was 17 kOe which is well beyond the anisotropy field (as calculated from the date of Darnell *et al.* [5] for chromium-modified Mn_2Sb) so that the samples were completely saturated magnetically.

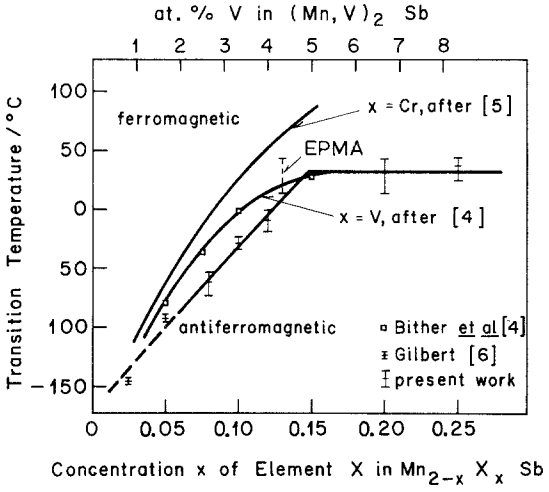

Figure 2 The transition temperature of vanadium- and chromium-modified Mn_2Sb [4–6] as a function of concentration together with the results from the present study. Also shown is the location of the vanadium solid solution limit in terms of the transition temperature observed in high vanadium alloys following EPMA analysis.