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SEM studies of fractures in spinodally hardened Cu–9Ni–6Sn–X alloys

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In a recent study [1, 2] it was shown that hardening by a spinodal decomposition process in Cu–9Ni–6Sn can be controlled to some extent by quaternary trace element additions. The added trace element did not have any influence on the spinodal decomposition process, but it significantly influenced the kinetics of the formation of the (CuNi) Sn precipitate. Because of the beneficial hardening observed in these alloys, the Cu–Ni–Sn spinodal alloys have come into commercial prominence in recent years [4, 5]. In the light of this, it was considered worthwhile to examine the fracture behaviour of these alloys in different thermally treated conditions.

Weighed quantities of copper, nickel and tin (of purity 99.99% or better) were induction melted and solidified into a button by centrifugal ejection into a graphite mould, 50 mm in diameter. In order to make the trace element addition, the desired element was also included in the initial charge. Alloys were prepared to contain 1% addition of any one of aluminium, iron, silicon, magnesium or manganese in the base alloy containing 9% nickel and 6% tin. A simple check was made by ultraviolet emission spectrography to confirm that the actual analysis was within 2 to 3% of the desired value for each element.

Each alloy button was homogenized in a vacuum of better than 10^{-5} Torr at 950° C and then rolled in one or more stages (depending on the alloy composition) to a final thickness of 1 mm. Tensile specimens (20 mm gauge length) milled out from these sheets were solutionized in a vacuum tube furnace at 950° C for 1 h, quenched in iced water and then aged at 390, 400 and 410° C. Hardening was monitored by measuring the Vickers' hardness number, VHN, as a function of the ageing period. In all cases the hardness gradually increased reaching a peak and thereafter decreased slowly. The rate of decrease in hardness was dependent on the alloy composition, it being the highest in the base ternary alloy. Based on the spinodal hardening curve for each alloy, testing in tension was carried out in peak-aged, under-aged and over-aged conditions. Fractured test specimens were immediately transferred into the test chamber of a S-410 Cambridge Stereoscan SEM and examined in the secondary emission mode. Results of fracture examination in a condition just beyond reaching the peak in hardness in each case have been reported in this communication.

In terms of elongation in the tensile test specimen the base alloy exhibited the lowest value [2], which further decreased with the ageing period. At an ageing temperature of 400° C the hardness reached a peak value in about 8 h and then continuously decreased in the base alloy. In all of the quaternary alloys, at 400° C ageing, after reaching a maximum value the hardness remained stable for a fairly long period of ageing [1]. In order to make the comparison of fracture behaviour more realistic, the quaternary alloys were aged for about 20% extra time after reaching the maximum in hardness, and then tested in tension.

Fractures in the base alloy exhibited regions of intergranular failure and the presence of large precipitate particles almost symmetrically separated from the matrix all around. This is indicative of failure by crack nucleation at the particle-matrix interface and interface deco-



Figure 1 Secondary electron image of fracture in the Cu–9Ni–6Sn alloy showing intergranular failure and particle-matrix interface decohesion around large rod/tabular shaped particles.

hesion (Fig. 1). It is also seen that a number of large sized rod-shaped particles appearing on the surface have fractured. A small number of dimples are also seen in some areas, but there is no evidence of much plastic deformation in the matrix in the interdimple region or on the dimple ridges. In comparison, fracture surfaces in the quaternary alloys in the averaged condition shown in Figs. 2 to 6 indicate a more ductile failure than that in the base alloy. This is especially so in the alloys containing iron, manganese and silicon additions. In alloys containing magnesium (Fig. 5) and aluminium (Fig. 6), however, the fractures appeared to be intergranular. However, on a careful examination at higher magnification, Fig. 6b, the fracture was observed to be transgranular. The appearance of Fig. 6a is because of the formation of a number of secondary cracks along the grain boundaries. In both the alloys there is no evidence for the

presence of large sized particles. In some of the quaternary alloy fractures, presence of dimples and particles in the dimple craters are noticable. The size of the particles is much smaller than those found in the base alloy after a similar ageing period. This clearly indicates that the precipitate particles in quaternary alloys have grown to a much lesser extent and are distributed more homogeneously in the alloy. Indirectly therefore, it may be inferred that the presence of the quaternary elements in the alloy has influenced the nucleation and growth of the precipitate particles. The exact mechanism by which this influence is brought about can be established only after determining the nature and composition of the precipitate in each alloy. In a more recent TEM study of these alloys, it has been confirmed [5] that the presence of the quaternary alloy influences the formation of the precipitate strongly, while it has no significant







Figure 3 Fractograph of the Cu-9Ni-6Sn-1Mn alloy with predominantly ductile dimples.



Figure 4 Fractographs of the alloy containing magnesium showing intergranular failure with microdimples on the facets.



Figure 5 Fractographs of the alloy containing silicon showing ductile failure.



Figure 6 Fracture in the Cu-9Ni-6Sn-1Al alloy showing a transgranular failure with strong cleavage markings.

influence on the spinodal decomposition process itself in the base alloy. (See the note below.)

The other observation concerns the geometry of the precipitates. In the base alloy the precipitates are large and possess rod-like and/or tubular shapes. In all the quaternary alloys the precipitates were globular in shape. In the alloy containing iron, however, the precipitates were thin and long, but much smaller in size than those in the base alloy (Fig. 2a). A close examination of the dimple ridges as well as inner dimple surfaces in these alloys reveals that a good deal of plastic deformation indented by such markings as slip, sharp wedge type failure and shear lips, respectively in the alloys containing manganese, silicon and iron (Figs. 2b, 3b and 5b) has taken place. In the case of the alloy containing magnesium, the presence of very fine dimples may be observed on the intergranular facets. On an over-all comparison of the fracture characteristics of the quaternary alloys with that of the base alloy, it may be observed that for the ageing conditions investigated, iron, manganese and silicon additions seem to impart improvements in the ductility of the alloy. However, this fact could not be confirmed from tensile test data, which is only limited in extent.

Results presented herein are clearly indicative of the control that can be exercised on precipitate size and distribution in the spinodal Cu-9Ni-6Sn alloy in the post-peak ageing period, and thereby achieving the property control. These quarternary alloys are now being studied in detail for their response to thermal and thermomechanical treatments by 'TEM, XRD and mechanical property measurements. *Note added in proof*: In a very recent XRD study by the authors of these alloys in terms of sideband formation, the presence of iron has been found to decrease significantly the spinodal wavelength.

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