than the previous data for amorphous Al_2O_3 [5, 6], which were in close mutual agreement, but this may be because a mixed oxide forms on heating or ion-beam thinning of the Al–Cu foils.

Williams and Edington estimated a cooling rate of $5 \times 10^8 \, K \, sec^{-1}$ from a eutectic lamellar spacing of ~20 nm, shown in their Fig. 2. This estimate is based on an assumed specimen thickness of ~150 nm whereas the area considered has been ion-beam thinned from an initially greater but unspecified thickness. We have emphasized previously [2] the need to consider the *as-solidified* specimen thickness t when estimating cooling rates $\dot{r}$ by this method since the derived $\dot{r}$ is sensitively dependent on t.

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The production of electron transparent areas by splat-quenching

Splat-quenching, using the gun technique [1], involves the use of a shock wave to impel molten metal droplets directly onto a cooled, inclined, copper substrate. The aim of the technique is to maximize the quench rate, thus giving rise to metastable, non-equilibrium structures. In the extreme case, areas thin enough for direct examination in the electron microscope at 100 kV are produced.

Recently, Vitek [2] proposed that the formation of such electron transparent areas was not simply due to the spreading of individual droplets upon impact with the substrate. Using combined scanning transmission electron microscopy (STEM) and scanning electron microscopy (SEM), two alternative mechanisms were proposed. These involved (a) the interaction on the substrate between a solidifying droplet and a subsequently impacting droplet that had undergone in-flight solidification, and (b) the interaction of a solidifying droplet with a flaw, or solid droplet, on the substrate. Regions arising from such droplet interactions will have undergone an unknown sequence of cooling and reheating, resulting in atypical precipitation reactions, since the specimens, in general, are highly supersaturated. It is the purpose of this communication to point out that, for comparative microstructural studies of splat-quenched specimens, it is essential that the regions observed have cooled under similar conditions. Furthermore, this situation can be approached in practice by manipulation of the experimental variables. It is possible to produce thin areas arising mainly from simple impact spreading. Such areas will have undergone a rapid, and most importantly, a relatively reproducible quench, thus permitting valid microstructural comparisons to be made between different specimens.

A thin region, similar to the type described by Vitek [2] is shown in Fig. 1. The sample of Al–17.3

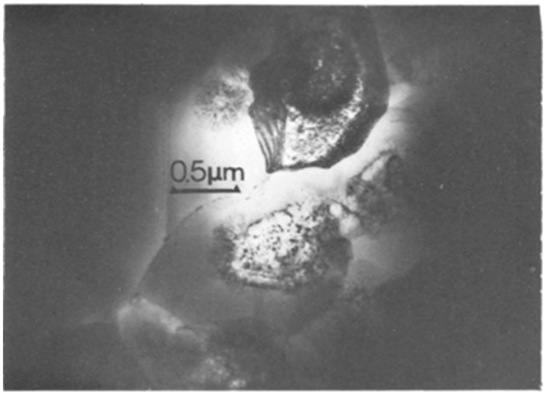

Figure 1 An electron transparent region surrounded by bulk material in a splat quenched Al–17.3 at.% Cu alloy.

Figure 2 An electron transparent region produced by single droplet spreading in a splat quenched Al–17.3 at.% Cu alloy.

at.% Cu was quenched under reduced pressure of argon in a modified Duwez gun system which has been described elsewhere [3]. The region is surrounded by bulk material opaque to 100 kV electrons, and thus it is impossible to characterize the temperature sequence responsible for the observed microstructure. In contrast, Fig. 2 shows a thin region at the edge of a hole in the foil. This region is surrounded by the bulk specimen (A) and a narrow band (B) of thicker material at the edge of the foil. Such a thickness distribution would arise through the spreading of a viscious solidifying droplet down an inclined surface, shown schematically in Fig. 3, which would be expected to occur during splat-quenching. It is considered that such regions alone should be used for comparative microstructural studies, in view of the relatively reproducible, rapid quench they have experienced.

The critical experimental variables controlling the production of areas like Fig. 2 were specimen mass and crucible nozzle diameter (the latter being the hole through which the molten metal is expelled by a shock wave of argon). In the present investigation of aluminium–copper alloys, it was found that specimens of mass 100 mg gave sufficient splat-cooled foil, whilst minimizing droplet overlap effects. Under this condition, the most efficient quench (in terms of thin area production) occurred using a nozzle diameter of ~0.85 mm. With smaller nozzle diameters, (<0.75 mm) the charge was not spread sufficiently on the substrate, although atomization was on a finer scale. With larger nozzles (>1 mm), the charge was expelled as several large droplets which cooled slowly, and also interacted with one another during solidification. The appropriate nozzle diameter will of course vary with melt superheat since the droplet viscosity will be affected, so the superheat should be kept constant. Similarly, some increase in area of droplet spread may be achieved by increasing the nozzle–substrate separation. However, this would result in greater in-flight cooling thus lowering the overall quench rate.

In conclusion, electron transparent areas may be produced in a variety of ways during splat-quenching, but the most useful, i.e. rapidly and reproducibly quenched, are produced by a single droplet spreading upon impact. It is possible to manipulate the experimental variables to optimize the production of such regions, thus permitting valid comparison of microstructures from different specimens.

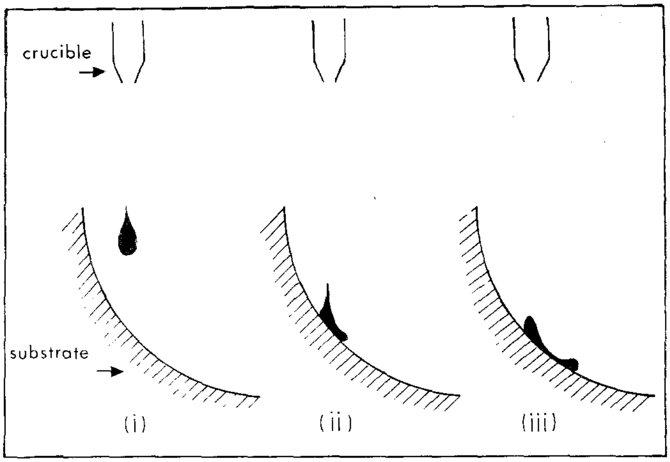

Figure 3 A schematic diagram illustrating the production of electron transparent thin areas by single droplet spreading.

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Comment on "The production of electron transparent areas by splat-quenching"

Recently, the author [1] had examined the conditions for the formation of two different electron transparent areas in a splat-cooled foil. The conditions were found to involve either droplet–droplet interactions or droplet–substrate interactions during quenching. The simple spreading of individual droplets into thin regions was not observed, but this mechanism was not eliminated as a possibility. Williams and Edington [2] have noted that the abundance of such thin areas can, to a significant degree, be controlled by varying the splatting conditions. In subsequent observations, the author has also found these "simple spreading" regions. Williams and Edington have also pointed out, as is likely to be the case, that because of the similar conditions under which they are produced, areas formed by the simple spreading mechanism have more reproducible microstructures than those produced through droplet-droplet or droplet-substrate interactions.

It is with regard to the reproducibility of the microstructures that I would like to add a few comments. In work dealing with amorphous Cu–Zr splatted alloys, thin areas presumably produced by simple droplet spreading (i.e. the thin areas were at the edge of the foil) were observed to have amorphous microstructures. However, some of these areas were also observed to have undergone partial crystallization during cooling. This would imply that within areas produced under similar conditions, the cooling rates varied significantly (the slower cooling rates resulting in partial crystallization). Unfortunately, a quantitative assessment of the difference in cooling rates is impossible to make. Nevertheless, it is clear that even similar solidification conditions may lead to significantly different cooling rates (and correspondingly different microstructures) and thus caution must be exercised when comparing the microstructures of gun-splatted foils.

In contrast, using the same Cu–Zr composition but a different splat-cooling technique (the piston and anvil technique [3]), more massive samples were obtained. For microstructural investigations, these "bulk" samples were electropolished. In these thinned foils, the microstructural features were more consistent than in gun-splatted, unthinned foils. By comparing areas within the same general location in the foil, only amorphous regions were observed, with no sign of partial crystallization. Naturally, cooling rates varied to some degree throughout the splats. However, those sections of numerous different splats in which the cooling conditions were optimal (as determined visually by the degree of oxidation, surface smoothness, etc) showed consistent, reproducible results in the form of completely amorphous microstructures.

Thus, in conclusion, although thin areas produced by a simple spreading mechanism in which the solidification conditions are similar probably have more reproducible microstructures than in areas produced by droplet–droplet or droplet–substrate interactions, even these areas need not have the same structural features. Probably the most reproducible and consistent microstructures can be obtained by thinning splat foils produced in a more massive form.