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COMPOSITION GRADIENTS THROUGH RIBBON THICKNESS OF THE MELT SPUN
AMORPHOUS $\text{Al}_{70}\text{Fe}_{13}\text{Si}_{17}$ ALLOY

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Abstract - The investigation of inhomogeneities through the ribbon thickness of an $\text{Al}_{70}\text{Fe}_{13}\text{Si}_{17}$ amorphous glass obtained by melt spinning has allowed to evidence the existence of composition gradients localized on the "wheel contact side" concavities, probably due to gas bubble trapping effects. The composition gradients were analyzed by XEDS in a scanning transmission electron microscope STEM on ultrathin ribbon sections prepared by ultramicrotomy.

I- INTRODUCTION

Experimental evidence of composition fluctuations through ribbon thickness of metallic glasses is rather rare /1/ although often assumed for interpreting crystallization behaviours /2/. In this study, ultrathin sections (ca. 100nm) of an amorphous $\text{Al}_{70}\text{Fe}_{13}\text{Si}_{17}$ alloy ribbon have been analyzed by X-ray Energy Dispersive Spectroscopy (XEDS) on a Scanning Transmission Electron Microscope (STEM).

II- MATERIALS and METHODS

$\text{Al}_{70}\text{Fe}_{13}\text{Si}_{17}$ amorphous ribbons have been prepared by melt spinning on a cleanned copper wheel under He atmosphere ; they were supplied by the "Laboratoire de Métallurgie de l'École des Mines de Nancy" /3/. Their section sizes are regular : 0.6mm width and 20 ± 2μm thick. Transverse ultrathin sectioning (fig.1) of the amorphous ribbon, embedded in a resin, was carried out by ultramicrotomy. For the XEDS analysis of the obtained thin foil, the electron probe diameter was 4nm at the entrance surface of the foil and because of the beam spreading through the specimen, the spatial resolution has been estimated to be 12nm /4/. The quantitative analysis of compositions was performed, for the beam positions indicated in fig.1, using the ratio method outlined by cliff and Lorimer /5/.

Fig.1 : Transverse ultrathin sectioning of the amorphous ribbon.
III- RESULTS and DISCUSSION

Figure 2 shows Al, Fe and Si concentration profiles for the beam scanned across the ribbon thickness. Both edges of this section have been previously identified as being representative of the wheel contact surface and external surface. The position of the analysis points along the thickness ribbon can be easily checked from the contamination spots due to each XEDS analysis. Moreover the sample thickness can be measured using the contamination spot method proposed by Rae et al (6) (fig. 3). In figure 2, strong Fe, Si and Al concentration gradients are observed at proximity of the "wheel contact side" on ca. 1.3μm. In figure 4, which is an enlargement of Fig.2, it is clearly shown that one set of composition gradients extends between pure iron on the wheel side and the nominal composition Al0.70Fe13Si17, while the second one, starts at a composition AlFeSi instead of pure iron. Various XEDS analyses have been performed along the wheel contact side parallelly to the ribbon edge: the iron concentration results are reported in Fig.2 ; they indicate that the composition gradients do not exist everywhere. It seems from Fig.2 that the iron segregation could be associated with surface irregularities as, for example, gas bubble trapping shown on the SEM (scanning electron microscope) image of Fig.5. As plotted in the AlFeSi ternary diagram of Fig.6, the different chemical composition variations obtained for both runs of the XEDS analyses of Fig.4, seem to fit an unique smooth curve joining the pure iron vertex and the nominal composition Al0.70Fe13Si17. In such a case, the strong variation of composition could be estimated to be somewhat concentric to the level of a concavity as proposed on the schema of Fig.5. We have checked by local diffraction that the ribbon is amorphous in the almost pure Fe segregation region, which is remarkable when considering that the amorphisable bulk composition is very narrow for that system. It would mean that the quenching rate is, even there, sufficiently high for enabling the formation of almost pure amorphous iron.

It seems also in Fig.2 that each element concentration exhibits after the first strong gradient, weak oscillations through the whole ribbon thickness. These composition fluctuations are significative as can be affirmed from those of Si, for which the measurement relative error is smaller than the fluctuation amplitude. It has also to be noted that the concentration oscillations occur around the average composition values of the ternary alloy. It is tempting to consider these oscillations as resulting from a "frozen spherical composition wave" generated at the gas bubble concavity centre.

Further experiments are needed to clarify the exact origin of these composition fluctuations. The strong iron enrichment on the wheel side could be understood in terms of a rejection of aluminium and silicon during freezing of this "hypereutectic" alloy, at least in the gas trapped regions where the solidification rate is low enough (Newtonian cooling). It does not appear in the regions of ideal cooling where the heat transfer is almost perfect. Such interpretation would be in agreement with the Davis et al (1) results, who have observed by Auger electron spectroscopy, segregations attributed to air trapping on the "wheel surface" of FeSiB amorphous ribbons.

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(3) J.M. DUBOIS, K. DEGHAN, Chr. JANOT and P. CHIEUX (1985) This conference
Fig. 2: Al, Fe and Si concentration profiles analyzed on the imaged ultrathin section. Both set of curves correspond respectively to both contamination spot lines.
Fig. 3: Contamination spots profiles developed on both surfaces of the thin foil (a) for a tilt of 45° (b) for a tilt of 0° (see ref.6 for details).

Fig. 4: Enlargement of fig. 2 of the wheel contact side edge.
Fig. 5: SEM imaging of both surface of an amorphous Al$_{70}$Fe$_{13}$Si$_{17}$ ribbon (a) external surface (b) wheel contact surface. (c) Schematism of the proposed concentric variation of concentration at the level of a cavity.

Fig. 6: Composition values of the both runs of Fig.2 reported the AlFeSi diagram.