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Transformation processes during annealing of Al-amorphous alloys

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Abstract

As the amorphous aluminum alloys represent the newest achievement in rapid solidification of Al-based high strength heat resistant materials, a study was undertaken on the amorphous alloys in the Al-RE-TM system, the rare-earth metal being a lanthanide mixture and the transition metal a Ni-Fe substitution in definite proportions. The decomposition on heating of the most highly alloyed amorphous alloy in the investigated series is characterized by differential thermal analysis, electron microscopy and X-ray diffraction.

General background

A new opening in the development of high-strength heat resistant light weight aluminium alloys has been brought about by the application of rapid solidification. The main strengthening mechanism in such "liquid quenched alloys" is the solid solution strengthening resulting from the formation of a supersaturated solid solution containing solute elements far above their maximum high temperature equilibrium solubility limit. The solute content can be made even higher if an amorphous alloy may be produced by rapid solidification, because the amorphous phase can be regarded to be a kind of highly concentrated solid solution which has a homogeneous atomic distribution and a favorable atomic configuration so as to reduce the strain resulting from supersaturation.

Although the liquid quenched crystalline Al-supersaturated solid solutions were well known since the very beginning of the rapid solidification development [1,2], giving rise to a new group of high strength aluminium alloys, -namely the Al-transition metals group-, the obtention of amorphous Al-based alloys is rather a new discovery. Amorphous Al alloys originate from the theoretical approach of the French researchers J.M.Dubois et G. Le Caer to define the amorphisation trend of metallic melts by a short-range order model governed by a local chemistry [3]. The principles of such a model extended to Al-alloys together with the results of the first successful experiments on an aluminium amorphous alloy (namely $\text{Al}_{80}\text{Cu}_{10}\text{Ni}_2\text{Mo}_2$) have been stated in a paper deposited in June 1982 to the Paris Academy of Sciences and published three years later. The ductile Al-Ni ternary amorphous alloys comprising either Si or Ge as an amorphisation non-metallic addition have marked in 1987 a new step. The latest and most interesting group of aluminium amorphous alloys intensively studied by the Japanese researchers [4] comprises a rare earth-metal (Al-RE systems) and an additional transition metal (Al-RE-TM systems). Our research is concerned with this new group of amorphous aluminium alloys.

Results and discussion

Two series of Al-RE-TM alloys have been prepared and then rapidly solidified by melt spinning at 30 m/s linear speed of the copper roller, 0.7mm distance between nozzle and copper roller surface and 785 mbar argon pressure. The compositions of the "as quenched" melt

Fig.1 Composition location of the studied Al-RE-TM amorphous alloys.

(a Mischmetall) whose atomic percent composition $\text{Ce}_{51}\text{La}_{22}\text{Nd}_{19}\text{PrSm}_2$ corresponds to an average atomic weight $A = 140.875$ g/atom g that is very close to the atomic weight of cerium. The present paper will be concerned with the characterization of the alloy lying at the confluence of the two series in fig. 1, that is the richest alloy both in transition metal and in rare-earth metal. In addition the examined alloy presents a partial substitution of Ni by Fe in a proportion of about 1/3.5 its precise composition in atomic percent being $\text{Al}_{82.4}\text{RE}_{9.3}\text{Fe}_{1.5}\text{Ni}_{0.8}$ or in a more convenient form $\text{Al}_{82.4}\text{RE}_{9.3}(\text{Fe}_{0.22}\text{Ni}_{0.78})_{1.5}$.

Fig.2 X-ray diffraction patterns of $\text{Al}_{82.4}\text{RE}_{9.3}(\text{Fe}_{0.22}\text{Ni}_{0.78})_{1.5}$ alloy, (Cu K_α), a) "as quenched" amorphous ribbon, b) crystalline equilibrium structure.

spun ribbons (≈ 30 μm thick and $\approx 2\text{mm}$ wide) have been determined by EDAX examination (Philips SEM 515 electron microscope equipped with X ray energy dispersive spectrometers) and they are indicated on the composition triangle of a ternary Al-RE-TM phase diagram (fig. 1). One series of these studied alloys lies within a narrow band of almost constant content in rare-earth metal (9.2-9.5 at%), while the second series lies within a band of almost constant content in transition metal (8.2-8.7 at%). The rare-earth metal in our alloys was a mixture of lanthanides

Fig.3 DTA curve for the amorphous alloy ($10^\circ\text{C}/\text{min}$, in vacuum).

The as quenched ribbon of the selected alloy was ductile and

hard, its Vickers microhardness tested at 10 gf load being 285kgf/mm². Such a high level for hardness is characteristic for the amorphous Al-RE-TM alloys and it originates from a strong attractive atomic interaction and from short range ordering [4].

The amorphous state of the ribbon was confirmed by X ray diffraction. The sharp Bragg peaks associated to crystallinity are completely absent in the diffraction pattern of the amorphous alloy (fig. 2a), while they are clearly manifest in the crystalline equilibrium structure of the same alloy (fig. 2b).

The changes in the structure of the alloy upon annealing have been investigated first by differential thermal analysis (DTA) and further by X-ray diffraction and scanning electron microscopy (SEM).

As seen from the DTA curve in fig. 3 two well defined exothermic peaks are manifest around 290°C and 325°C indicating that the amorphous alloy decomposes into a more stable state in a limited temperature range. In order to clarify the structural changes due to the exothermic reactions, X ray diffraction analyses were made after an 1 hour annealing applied to the amorphous alloy at different temperatures: at 250°C (before the first exothermic peak), at 300°C (before the second exothermic peak) and at 350°C (after the second exothermic peak). As seen from the X-ray diffraction patterns in fig. 4a the amorphous state is not changed by 1 hour annealing at 250°C. The X ray diffraction pattern in fig. 4b shows that some crystallization has occurred during the first exothermic reaction at 290°C. Finally the X-ray diffraction pattern in fig. 4c (recorded after 1 hour annealing at 350°C) shows that a more intensive crystallization has occurred during the second exothermic peak at 325°C; indeed peak positions and intensities are almost the same as for the equilibrium structure (fig. 2b).

Fig.4 X-ray diffraction patterns recorded with Cu K_α radiation for the heat treated amorphous ribbons after 1h annealing at the specified temperatures.

The crystallized structure obtained after 1 hour annealing at 350°C has then been examined at high magnification in a SEM electron microscope (fig. 5a,b). A heterogeneous structure is manifest showing a high proportion of fine particles rich in the heavy elements (RE and TM) -the white regions-. These extremely fine particles (0.1-0.5 μm) consisting in Al-RE-TM intermetallic compounds are embedded in the aluminium rich matrix (the black regions), the overall aspect being typical for a dispersion hardened structure. For sake of comparison the electron micrograph and the Al X-ray distribution image for the slowly solidified alloy are also presented. It is obvious that the equilibrium structure (fig. 6a,b) is hundreds of times coarser than the structure obtained by amorphization followed by crystallization.

Fig.5 SEM micrographs of melt spun ribbon of $\text{Al}_{82.4}\text{RE}_{9.3}(\text{Fe}_{0.22}\text{Ni}_{0.78})_{8.3}$ alloy after $350^\circ\text{C}/1\text{h}$ argon annealing showing the decomposition crystallization of the amorphous solid solution:

a) 4250 magnification;

b) 23500 magnification.

Fig.6 Equilibrium microstructure of slowly solidified

$\text{Al}_{82.4}\text{RE}_{9.3}(\text{Fe}_{0.22}\text{Ni}_{0.78})_{8.3}$ alloy:

a) SEM micrograph;

b) X-ray image (Al K_α) showing aluminium distribution.

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