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TEM study of Discontinuous Reactions in Highly Supersaturated Cu-Co alloys

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Discontinuous precipitation (DP) reactions are solid-solid phase transformations taking place during aging of supersaturated solid solutions in conditions in which the precipitation process is driven by diffusion along moving grain boundaries. In Cu-Co alloys the precipitate products are formed as Co rod-like colonies, growing in a cooperative fashion and with diameter and spacing in the order of few nanometers [1]. This reaction has been extensively studied in innumerous systems for being related to degradation of the microstructures of precipitation-hardening alloys, thereby affecting its mechanical and physical properties. In Cu-Co alloys, DP has not called attention since it occurs in considerable smaller volume fraction when compared with other systems, leading to a very small influence in final properties of the material. Nowadays, with the development of spintronic and the use of Cu-Co alloys in sensors and spin-valves, one can expect that, given the fact these rod-like Co colonies generated by DP grow with rod diameter and interspacing in the order of the thickness of Cu-Co multilayer [2], it could drastically influence the giant magnetoresistance (GMR) ratios exhibited by this alloy system.

In this research we have studied the occurrence of discontinuous reactions (DR) in melt-spun Cu-10at.% Co aiming at evaluating its influence in GMR ratios. Melt-spun samples with average grain size of 1μm were isothermally aged at different temperatures for different periods of time resulting in well-defined volume fractions of DR. The microstructural characterization was performed in a TEM/STEM JEOL JEM 2100F instrument operating at 200kV under diffraction contrast mode and analytical electron microscopy using Thermo SEVEN energy dispersive X Ray spectrometry (EDX). Nano beam diffraction mapping NANOMEGAS technique has been used to evaluate orientation relationships between precipitate colonies/parent matrix as well as preferential directions of discontinuous reactions upon grain boundary migration.

Results showed that the as-solidified alloy was formed with columnar grains structure, a recognized morphological feature of this mode of rapid solidification. As can be seen in Fig1a, a bright field image in plain view after 5min of aging the grain growth was followed by DP in all the grain boundaries. In the sequence of aging, upon boundary migration, DP colonies exhibit a coarsening effect: the fine rod structure is replaced by wider inter-spaced Co-rich aggregates, driven by the grain boundary reaction front, thereby reducing the interfacial energy (Fig1b and c), in a process referred as discontinuous coarsening (DC). Contrary to the general case in several systems, where DC occurs associated to the mutual impingement of growing DP colonies, in this system we see that DC takes place while the parent matrix is not consumed by DP products. EDX elemental mappings of DP and DC are shown in Fig.2. An interesting feature verified was the bulging of grain boundaries by DC, as can be seen in Fig.3a and b. Based on this study we can conclude at this stage that grain boundary migration is driven by an excess of super saturation across the grain boundary reaction front, in a process described by Balluffi and Cahn [3] as chemically induced grain boundary migration (CIGM). The coarsening driving force is the minimization of interfacial energy between Co rod and Cu depleted matrix and the process stops when the super saturation is consumed.

GMR ratios were evaluated using a vibrant sample magnetometer operating at room temperature for fields up to 25000 Oe. Results are illustrated in Fig.3c, presenting an oscillatory behavior dependent on the aging time suggesting an interaction of the magnetic moments of the precipitates.

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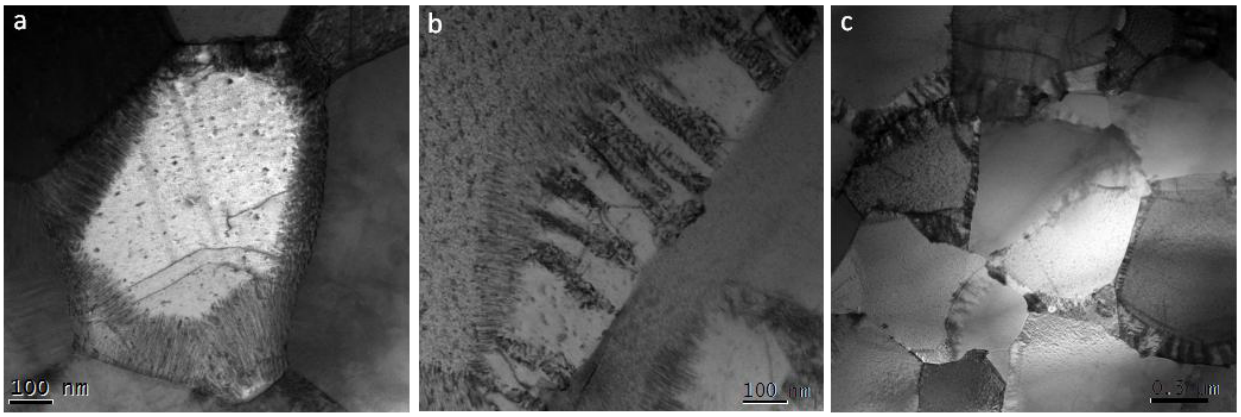

Figure 1 – Discontinuous reactions in Cu-10%Co. (a) STEM/BF of discontinuous precipitation in a sample aged for 5min; (b) Discontinuous coarsening after aging for 30min; (c) Overview after aging for 60min showing coarsening taking over in all grain boundaries.

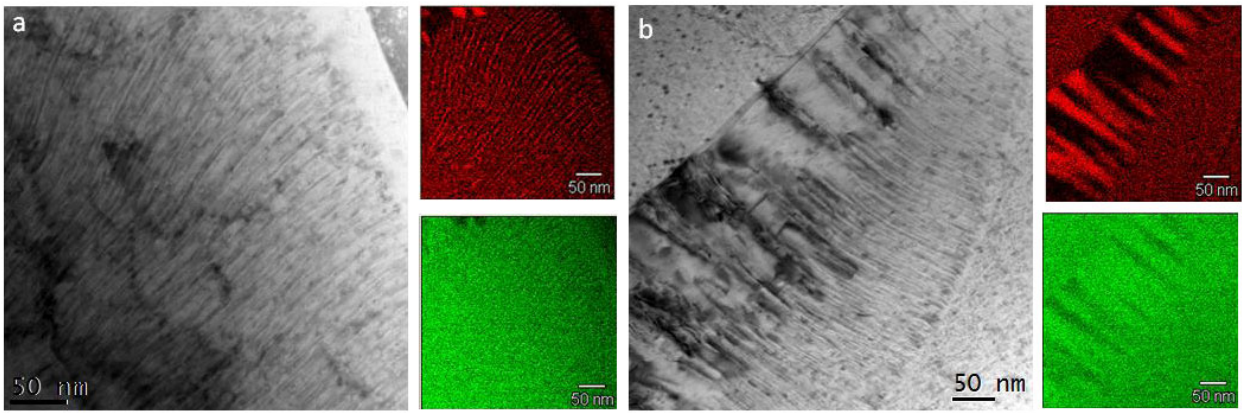

Figure 2 – BF/STEM and elemental mapping of (a) 5 and (b) 30min aged samples presenting DP and DC respectively. Co mapping is in red and Cu in green.

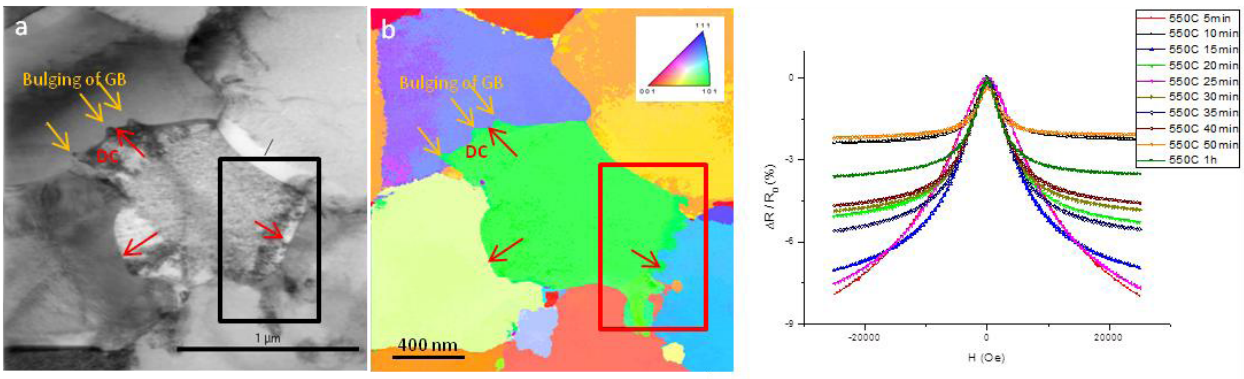

Figure 3 – Nano beam diffraction of a sample aged for 30min; (a) Bright field reconstructed image and (b) orientation map. (c) GMR ratios as function of the applied field.

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