



	Experiment title: In situ kinetic analysis of the formation of quasicrystalline phases on Al based thin films	Experiment number: HS-443
Beamline: ID1	Date of experiment: from: 06 may 1998 to: 12 may 1998	Date of report: 27 aout 1999
Shifts: 18	Local contact(s): Jean-Luc Joulaud	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): C. Bergman*, CTM/CNRS 26 rue du 141eme RIA 13331 Marseille P. Gas*, E. Emeric*, Metallurgie/CNRS, Fac. Saint Jerome, Cas 511, 13397 Marseille T. Grenet*, F. Giroud*, C. Loubet*, LEPES-CNRS BP 166 38042 Grenoble cedex 9 J. L. Joulaud*, M. J. Capitan*, ESRF, BP 220, 38043 Grenoble cedex		

Report:

Thin films of quasicrystals (QC) can be prepared by heat treating metallic elemental multilayers. The study of the reactions occurring during the heat treatments is important, first because it allows a study of the thermodynamics and kinetics aspects of the formation of QC phases by solid state reactions, second because it permits to optimize the conditions of fabrication of thin film samples which are of importance for applied as well as fundamental studies. The purpose of the project was to study the transformations occurring during the annealing of multilayers. Two systems were studied: (Al,Cu) and (Al,Cu,Fe). The present report deals with the second system (the studies on the first system are the subject of another report).

In the (Al,Cu,Fe) system, two kinds of multilayers were studied, characterized by their stack sequence: Al/Cu/Fe/Cu/Al (ACFCA) and Al/Fe/Cu/Fe/Al (AFCFA) of total thickness 6000 Å. It was previously suggested that to get the pure $i\text{-Al}_{62.5}\text{Cu}_{25}\text{Fe}_{12.5}$ films, one should use the second sequence in order to avoid the formation of stable (Al,Cu) binary alloys and to start forming the Al_3Fe alloy, a possible precursor of the QC (Cu and Fe are immiscible at moderate temperatures). In order to test this hypothesis, diffraction spectra were recorded

during slow (2°C/min) ramp annealings up to 700°C in order to identify the different phases successively formed.

ACFCA sequence: the first phase to form at around 200°C was the Al₂Cu phase, leaving some unreacted Al. At 350°C, one observed the disappearance of the Al₂Cu and the formation of AlCu, along with the total consumption of the remaining free Al. Although surprising, this can be explained by a reaction involving the Fe layer which was consumed, forming an Al rich alloy. Unfortunately the latter could not be identified unambiguously. At higher temperatures, ternary phases were observed (ω and β phases), which finally were consumed as the QC appeared (starting from 550°C). These results showed that pure QC films can be prepared starting from the ACFCA sequence, with the prior reactions forming (Al,Cu) binary alloys. No indication of the formation of Al₃Fe was observed.

AFCFA sequence: surprisingly the same main phases as for the other sequence were observed. The Al₂Cu peaks appeared above 300°C, then the AlCu peaks and those of ternary phases, until the quasicrystal formed and remained alone at the highest temperatures. The AFCFA sample a priori possesses no Al-Cu interface. Cross sectionnal TEM pictures performed on similar samples showed continuous compact Fe layers separating the Al and Cu ones. The fact that the (Al,Cu) binaries formed at higher temperatures than in the ACFCA sample shows that the Fe layer acted as a diffusion barrier between Al and Cu, but did not take part in the reactions in the first steps. In both sequences, Fe only takes part in the transformations once Cu has already reacted with Al.

These experiments have shown that whatever the stack sequence of the multilayers, pure QC films are obtained upon annealing, via the formation of (Al,Cu) binary alloys as the first steps, and that the expected Al₃Fe phase never appears. They have opened the way to further studies. In particular complementary TEM studies of the phases successively formed in the two sequences are underway. A paper was submitted to the ICQ7 Conference, and another one is in preparation.