| ESRF | **Experiment title:** In-situ investigation of the solid-state reaction reasulting in the formation of quasicrystals in mechanically alloyed Al-Cu-Fe alloys | **Experiment number**: HS-857 |
|---|---|---|
| **Beamline**: BM16 | **Date of experiment**: from: 24 June 1999 to: 27 June 1999 | **Date of report**: |
| **Shifts: 12** | **Local contact(s)**: I. Pape, A. Fitch | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

*Dr A.M. Korsunsky[1], Dept. MMME, Univ. of Newcastle, NE1 7RU, UK

*Dr A.I. Salimon, X-ray Physics Dept, Moscow Inst. for Steels and Alloys, Russia

*Dr A.M. Polyakov, X-ray Physics Dept, Moscow Inst. for Steels and Alloys, Russia

[1]Present address: Department of Engineering Science, University of Oxford, Parks Rd, Oxford OX1 3PD, UK


**Report:** Owing to their unusual lattice structure and a unique combination of mechanical and thermal properties, quasicrystalline materials are considered as attractive candidates for a number of structural applications. Obtaining these materials in the desired form economically presents a complex technological problem. In the course of our recent investigations we have found a way to overcome most of the diffriculties by using a combination of mechanical alloying / powder metallurgy approaches. Our preliminary X-ray laboratory studies revealed that a series of solid-state reactions unfold during annealing of mechanically alloyed Al-Cu-Fe powders, and these result in the formation of effectively pure and stable quasicrystalline phase. Several metastable, and often not sufficiently well-identified, intermetallic compounds form in the solid-state reactions. Their decomposition and interaction lead to the appearance of stable F-type quasicrystals. Our main goal in the present experiment was to apply the advantages of the high brilliance synchrotron X-ray radiation delivered by the ESRF with the simulated annealing sample environment, thereby being able to monitor the solid-state transformations *in situ* and in real time with the highest possible resolution and reproducibility.

This aim has been largely achieved, allowed several important tasks to be addressed which are essential for all-round characterization of the solid-state interactions in the Al-Cu-Fe system. These are:

- X-ray patterns with excellent statistics (Fig.1) suitable for further Rietveld refinement and quantitative phase analysis were collected;
- The temperature regions of specific solid-state reactions were determined with higher precision. This is particulary important for the characterisation of the structure transformation from the primitive type to the F-type of icosahedral quasicrystals (Fig.2). The analysis of this transformation is of great theoretical importance; however, this transformation remains relatively poorly discussed in the literature;
- The temperature region 550-750°C was investigated in some detail, and the approximant-to-quasicrystal transition was studied at these temperatures. The thermal relaxation was studied of line broadening due to phonon and phason type defects present in the growing quasicrystalline phase;
- The characteristic times of the transformations were evaluated, making it possible to describe the kinetics of the corresponding solid-state reactions.
- Since the transformations taking place during annealing are irreversible, the data collected during cooling of the fully transformed powder from 850°C to room temperature contained all the necessary information for the precise calculation of the linear thermal expansion coefficient of the icosahedral quasicrystal. The variation of this parameter can

be determined practically up to the melting temperature (about 850°C), and the experimental data about thermal behaviour of this physical property may be succesfully compared with theoretical predictions;

- It was established in the course of the experiment that small variations in the chemical composition of the quasicrystalline phase led to noticeable changes in the relative intensities of the main peaks. These differences are related with the variation in the lattice structure and prefential atomic sites occupancy.

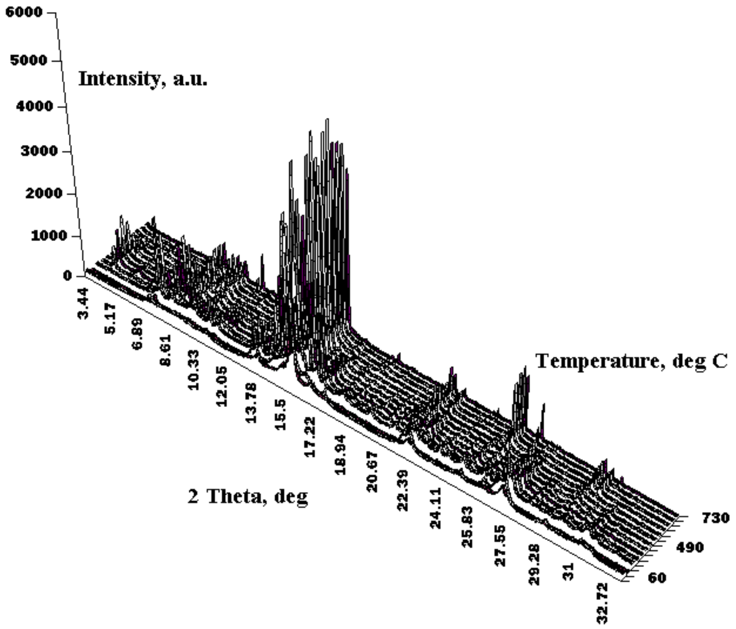


Fig. 1 Temperature changes in X-ray patterns of mechanically alloyed Al-Cu-Fe alloys.

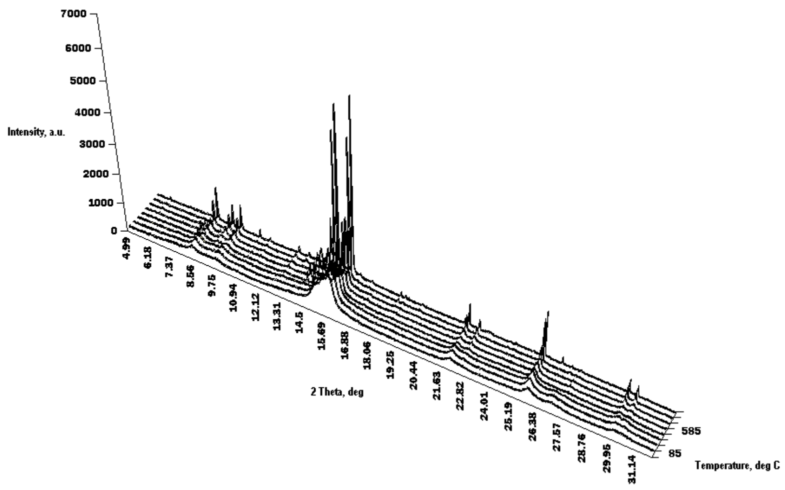


Fig.2 Transformation of the primitive-type to F-type icosahedral quasicrystal.

The latter effect which we observed clearly suggests a method for atomic structure characterization and description, compared to the complexity of applying 6-dimensional crystallography methods. We elaborated a computer programme for the modelling of powder diffraction patterns based on the information about Ammann rhombohedra decoration. As we demonstrated in [1], this programme succesfully applies to the modelling of primitive (Al-Mn type) quasicrystals. However, the same procedure for icosahedral F-type quasicrystal, such as Al-Cu-Fe one, leaves some small, but unacceptable discrepancies, which are due to the lack of precise data about atomic site occupancy by different atomic species. This obstacle, however, may be overcome by undertaking further experimental study of quasicrystalline powder diffraction patterns. In particular, the use of radiation with wavelenght just below and above the absorption edge of the elements constituting quasicrystals seems promising. Since the atomic scattering factor changes dramatically under such conditions, drastic changes in the intensity of certain reflections are expected. This effect is well-known for ordered intermetallics, and can be quantitatively predicted by our computer programme for the quasicrystals constructed out of Ammann rhombohedra. A new proposal dedicated to this type of experiment is in preparation.

**[1].** A.I. Salimon, A.M. Korsunsky, E.V. Shelekhov, T.A. Sviridova. Mater. Sci. Forum, Proc. of EPDIC-6, 22-26 August, 1998.