

AlCr quasi-crystalline thin films

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Recibido el 9 de mayo de 1995; aceptado el 26 de junio de 1995

ABSTRACT. Concentrations and substrate temperatures which yield the icosahedral phase in multilayered thin films of AlCr evaporated onto heated substrates were found. The thickness of the different layers was controlled to yield the desired atomic percent. The presence of the icosahedral phase was determined by electron diffraction via TEM and the crystalline directions for each ring identified. The icosahedral phase is found at about Al 14 at. % Cr with substrate temperature of the order of 350°C.

RESUMEN. Se hallaron las concentraciones y temperaturas del sustrato que permiten la formación de la fase icosaédrica en películas delgadas multicapas de AlCr evaporadas sobre sustratos calientes. Se controló el espesor de las diferentes capas de manera que se logró el porcentaje atómico deseado. La presencia de la fase icosaédrica se determinó mediante difracción de electrones con microscopía electrónica de transmisión (TEM) y se identificaron las direcciones cristalinicas correspondientes a cada anillo. Se halló la fase icosaédrica a concentraciones alrededor de Al 14 at. % Cr con temperaturas del sustrato del orden de 350°C.

PACS: 61.16.Di; 61.42.+h; 68.65.+g

Schechtman *et al.* [1] opened a new chapter in the study of materials discovering the icosahedral phase in Al-Mn. This phase shows long range orientation order without translation symmetry and is described by the group $(m\bar{3}5)$. However the icosahedral phase in Al-Cr (discovered also by Schechtman), has received little attention [2,3]. De Lima *et al.* [4] worked with multi-layers of Al-Cr but did not study this phase. Bancel *et al.* [5] report electron microscopy and HR x-ray scattering on Al₆Mn. They index the x-ray peaks to a mixture of fcc Al and a sum of icosahedral vectors.

The author with some colleagues have reported [6] a convenient way to produce the icosahedral phase in Al-Cr, which was determined by the presence of the rings in the diffraction pattern of the samples. After further study, the data confirm the presence of the icosahedral phase in such films as will be described, and the crystallographic directions are identified.

The samples were prepared as thin films of successive layers of Al and Cr, e-gun evaporated at 2×10^{-6} Torr, onto TEM grids covered by a carbon membrane. Thicknesses are chosen to yield the desired at.%. Substrates were heated to the desired temperature [6] (Table I). Once the evaporation is completed, the final concentration is calculated from the thickness of the five different layers. Electron diffraction patterns of the samples were obtained by 100 KeV electrons from a H 7000 TEM. Rings diameters were measured and

TABLE I. AlCr thin films alloys data. Total thickness (t) for each metal and average temperatures (T) during evaporation time.

| Al at.% Cr | t Al (nm) | t Cr (nm) | T °C |
|------------|-------------|-------------|--------|
| 12.11 | 93.3 | 9.3 | 348 |
| 13.12 | 92.5 | 10.1 | 351 |
| 14.07 | 91.2 | 10.8 | 364 |

normalized to the [111] Al ring. Table II is a partial reproduction from [6] with the new information added. Errors in the diameters ratios are less than 2%, error in concentrations is less than 0.1% and thermocouples errors are in the order of 0.2°C, but during deposition the temperature varied in the order of 10°C about reported average temperature (Table I).

Table II shows the ring diameters normalized to the most intense Al ring, (111). Data from Ref. [6] for Al, Al 12.1 at.% Cr, Al 13.1 at.% Cr and Al 14.1 at.% Cr are reproduced, with a few data reallocated in the light of the new information available. The last two columns were obtained from Ref. [5] in the following way: the x-ray diffraction pattern in their Fig. 1 was worked in order to obtain the ratios of their Q's to that of Al (111), the corresponding crystallographic index appears in the last column.

Figure 1 shows the electron diffraction pattern for Al 14.1 at.% Cr deposited at about 364°C and its diagram. One can observe the icosahedral phase rings (dashes in diagram) as well as some Al rings. The figure also shows the polycrystalline nature of the sample because the rings are a set of points; however, the selected area includes many crystallites.

From Table II one can see that our samples present the icosahedral phase, and even though some Al rings appear, they are weak (see figure). The new contribution in this paper is the identification of the crystallographic directions for these multi-layered thin films and the way they were obtained. Besides it is clear that icosahedral AlCr has the same structure as icosahedral AlMn.

All Al rings in Al 12.1 at.% Cr are smaller than those of pure Al, this means larger interplanar distances. Being the radius of Cr larger than that of Al one could think that at least part of Cr enters substitutionally in the Al lattice. However, the presence of strong rings which ratios agree well with the ratios of the icosahedral phase x-ray peaks show that this phase is present.

For Al 13.1 at.% Cr on the other hand, Al rings are larger (and weaker [6]). This composition shows more icosahedral rings and they are stronger than for Al 12.1 at.% Cr. It can be thought that the fcc Al is pressed by the advance in formation of the icosahedral phase. Rings with ratios 1.54, 2.14 and 2.59, not shown by the other compositions but present in Ref. [5], appear.

For Al 14.1 at.% Cr no general comment can be made when comparing with the rings from column 1; of those present some are larger and some smaller than those of pure Al, but all are weaker. The icosahedral rings appear more clearly than in the other samples. Rings with ratios 0.38, 2.01 and 2.21 not shown in Ref. [5] appear. They could belong to the precipitant phases. The absence of rings for some icosahedral indexes can be explained in part because they are very weak and could not be observed in the photos, for instance (200000), see Fig. 1 from Ref. [5]; or because they way our samples were produced induces

TABLE II. Rate of rings diameters to the most intense Al peak for this thin film alloy system. Columns show concentrations, average substrate temperature, ratio of peaks in Bancel *et al.* and crystallographic index.

| Al | 12.1 at.% Cr 348°C | 13.1 at.% Cr 350°C | 14.1 at.% Cr 364°C | ratio from [5] | index |
|------|-----------------------|-----------------------|-----------------------|-------------------|---------|
| | | | 0.38 | | |
| | | 0.59 | 0.60 | 0.61 | 110001 |
| | | | | 0.7 | 1110-10 |
| | 1.03 | 1.07 | 1.05 | 1.07 | 100000 |
| | 1.11 | 1.13 | 1.12 | 1.13 | 110000 |
| 1.15 | 1.14 | 1.16 | 1.18 | 1.15 | 200 |
| | | | | 1.31 | 210001 |
| | | 1.54 | | 1.55 | 111000 |
| | | | | 1.59 | 111100 |
| 1.63 | 1.60 | 1.64 | 1.63 | 1.63 | 220 |
| | 1.82 | 1.82 | | 1.82 | 101000 |
| 1.91 | 1.89 | 1.92 | 1.88 | 1.9 | 311 |
| 2.00 | 1.98 | | 1.97 | 1.99 | 222 |
| | | | 2.01 | | |
| | | 2.14 | | 2.13 | 200000 |
| | | | 2.21 | | |
| 2.32 | 2.29 | | 2.33 | 2.29 | 400 |
| 2.53 | 2.50 | | | 2.5 | 331 |
| 2.60 | 2.56 | 2.55 | | 2.56 | 420 |
| | | 2.59 | | 2.61 | 211100 |
| 2.84 | 2.81 | 2.89 | 2.87 | 2.8 | 422 |
| 3.03 | 3.00 | | 3.08 | | |
| 3.30 | 3.27 | | | | |
| 3.45 | 3.44 | | | | |

growth in some preferred directions, for instance the ring for (111000) is present only in Al 13.1 at.% Cr.

It is worth mentioning that we obtain the phase with thicknesses an order of magnitude larger than Levi and Schechtman [3]. We believe that this is possible because, being the substrate hot, the deposition and interdiffusion are simultaneous processes, whereas for [3] the phase itself acts as a barrier to interdiffusion. Thus our procedure to prepare the sample is more efficient and simpler since it needs no annealing after deposition.

Concluding, the icosahedral phase in AlCr was obtained in samples prepared by a simple procedure, the device has been explained in Ref. [6], which can be built and used in most laboratories and is able to offer samples of a variety of shapes. The crystallographic directions were identified for the icosahedral rings with the help of the paper by Bancel *et al.* [5]. This work is being continued with other concentrations and substrate

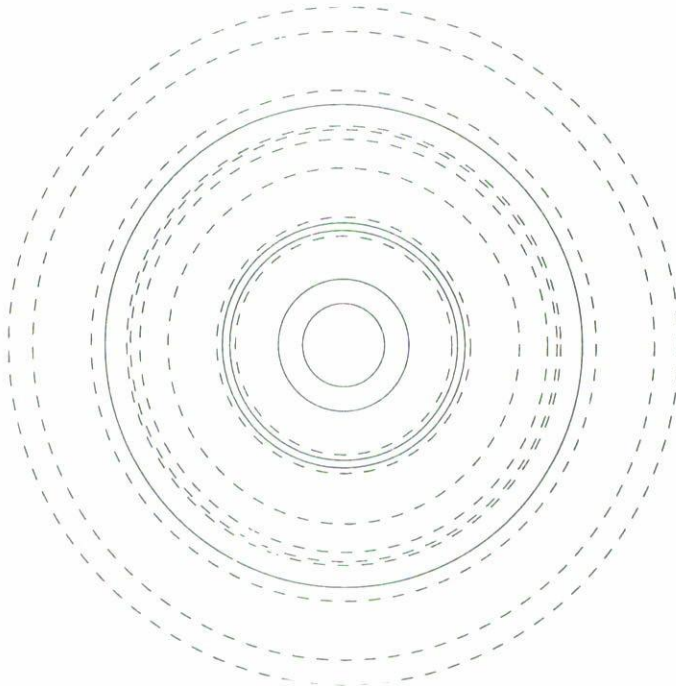
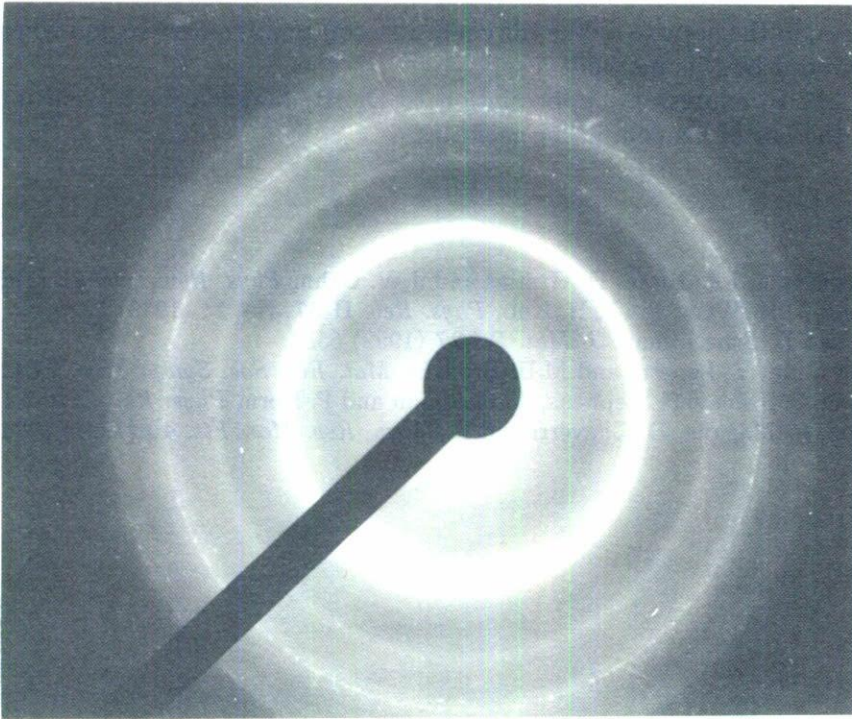


FIGURE 1. Diffraction pattern and diagram for Al 14.1 at.% Cr evaporated over substrate at about 364°C, some Al rings are present but those of the icosahedral phase are very clear (dashes).

temperatures, in the hope to define the concentration-temperature region where this phase can be produced by this method.

I would like to express my appreciation to Dr. R. Magaña for his encouragement as well as for the use of his computing facilities.

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