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Preparation and study of Al–Cr, Al–Cr–Mn quasicrystalline powders

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The production technique of Al–Cr, Al–Cr–Mn alloy quasicrystalline powders and their morphology, composition, diffraction pattern and thermal stability were studied. Computer processing of elevated-temperature X-ray diffraction data, enabled kinetic phase transformation diagrams to be plotted. It was shown that in Al–Cr, Al–Cr–Mn alloys, the composition and cooling rate dramatically affect the formation of the icosahedral phase; at a cooling rate of 10^5–10^6 K s^{-1}, nearly full icosahedral phase $Al_{82}Cr_{18}$, $Al_{82}(Mn, Cr)_{18}$ powders could be produced. For Al–Cr alloy, the thermal stability of quasicrystalline phase is improved with the increasing chromium content. The addition of the third constituent, manganese, can also improve the thermal stability of quasicrystalline phase.

1. Introduction

Since the discovery of quasicrystalline phase in rapidly solidified Al–Mn alloy by Shechtman *et al.* in 1984 [1], much research has been done on the quasicrystalline state of Al–Mn alloy. In comparison with the Al–Mn system, Al–Cr quasicrystals are more difficult to form, and consequently there are fewer studies reported on Al–Cr quasicrystal, and many problems remain unsolved. The preparation technique of Al–Cr and Al–Cr–Mn alloy quasicrystalline powders and their composition range, diffraction patterns, thermal stability as well as phase transformation during the crystallization process were studied and the results are reported here.

2. Experimental procedure

The quasicrystalline powders used in this investigation were produced using a rapid solidification device designed by the authors [2] in which the cooling rate reaches 10^5–10^7 K s^{-1}. As shown in Fig. 1, metallic melt was first atomized with a nozzle, then immediately, the droplets were centrifugally pulverized by a high-velocity rotating cone; subsequently they fell on to a high-velocity rotating disc, were cooled by the disc and the jetted cooling agent, and were converted to quasicrystalline powder. Aluminium, chromium and manganese of purity greater than 99.5% were used. Molten alloys at temperatures 150–200 K above their liquidus temperatures were rapidly solidified to form powders, their average particle size was 17–20 μm.

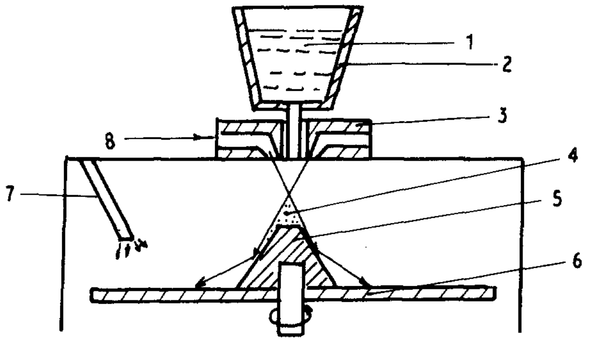


Figure 1 Schematic drawing of the rapid solidification device for making quasicrystalline powder.

3. Results and discussion

3.1. The effect of alloy composition and cooling rate on the formation of quasicrystalline phase

The composition and cooling rate dramatically affect the formation of icosahedral phase (I phase) when the rapid solidification technique is used to produce a quasicrystalline powder. For Al–Cr alloys, when the chromium content is less than 10 at %, the powder produced consists mainly of α-Al phase as well as I phase; when the chromium content is increased to above 15 at %, the amount of α-Al phase decreased drastically, and conversely, the amount of I phase increased appreciably and a new phase (X phase) appears. When the chromium content is higher than 18 at %, α-Al phase disappears, I phase increases and X-phase maintains the same level as in $Al_{85}Cr_{15}$ alloy. Increasing cooling rate can result in nearly full I phase powders; another new phase, Y phase, appears for 25 at % or higher Cr content, otherwise, amorphous powder forms when the cooling rate is increased. Fig. 2 shows the effects of composition on the formation of Al–Cr quasicrystalline powders at the same cooling rate. For Al–Cr–Mn alloy, the increment in both chromium and manganese contents increases the amount of I phase and decreases the amount of α-Al phase. As shown in Fig. 3, for the formation of I phase, an increase in manganese content is more effective than an increase in chromium content; furthermore, no X phase which exists in the Al–Cr quasicrystal can be detected in Al–Cr–Mn alloy. Cooling rates also affect quasicrystal formation in the Al–Cr–Mn alloy; at a lower cooling rate, full I phase powder can be obtained when the total Mn + Cr content is not less than 22 at % and when manganese dominates. In contrast, at higher cooling rate, only an Mn + Cr content of more than 18 at % can result in full I phase powder.

3.2. Thermal stability of quasicrystalline powders

Elevated-temperature X-ray diffractometry was used to study the thermal stability of Al–Cr quasicrystalline powders and their phase transformation during the crystallization process. The following results were obtained.

Al–10 at% Cr alloy:

$$(\mathrm{RT}-623\,\mathrm{K})\ \text{fcc Al} + \text{qcI} \xrightarrow{623-823\,\mathrm{K}} \text{fcc Al (decreasing)} + \text{qcI (decreasing)} + \text{X phase (increasing)} \xrightarrow{823\,\mathrm{K}} \text{X phase (maximum)} \xrightarrow{823-1073\,\mathrm{K}} \text{X phase (decreasing)} + \mathrm{X_1}\ \text{phase (increasing)} \quad (1)$$

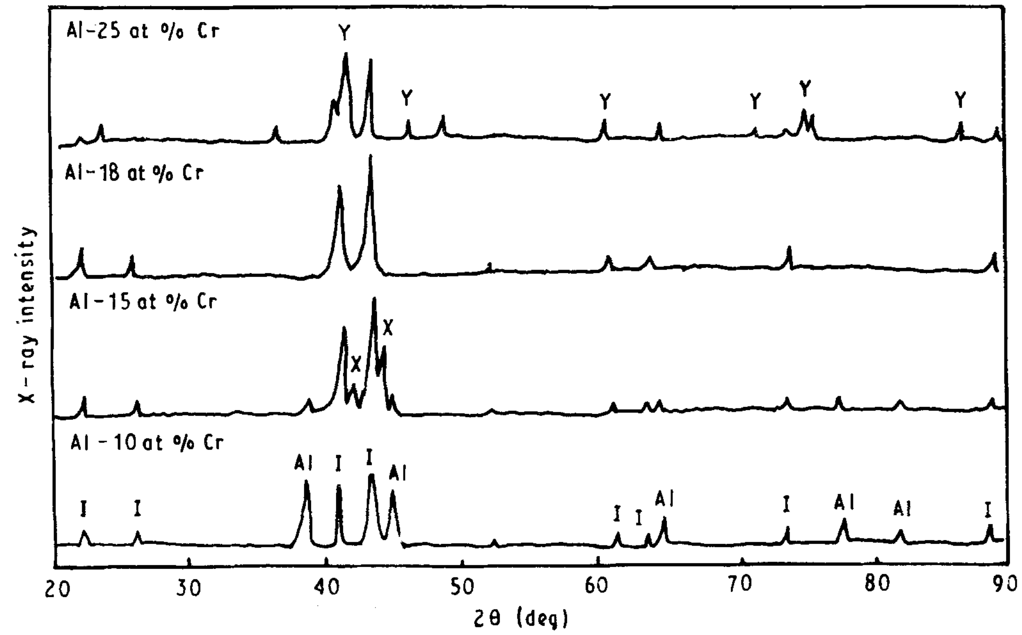
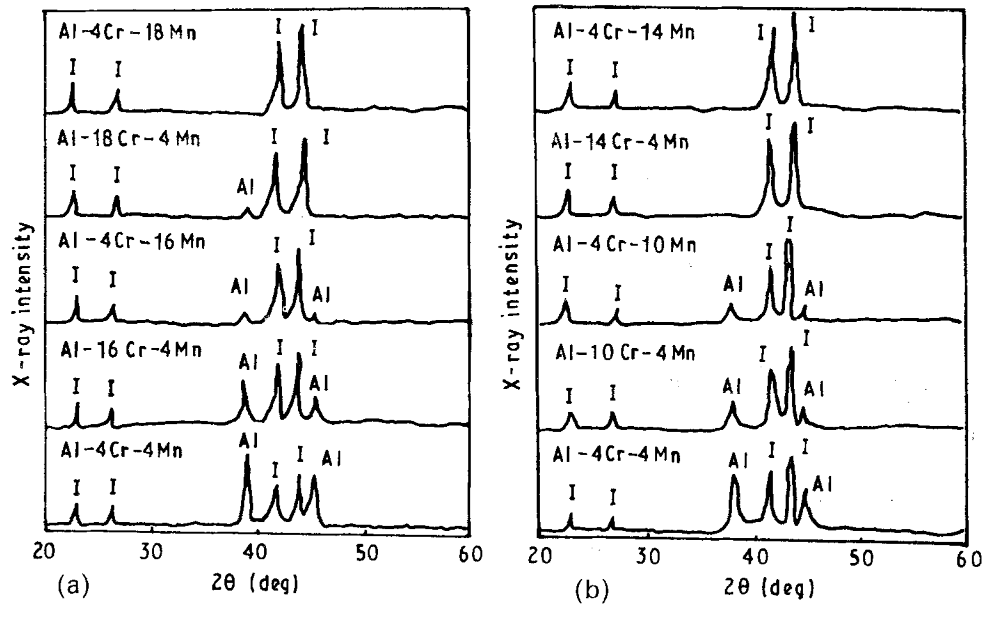


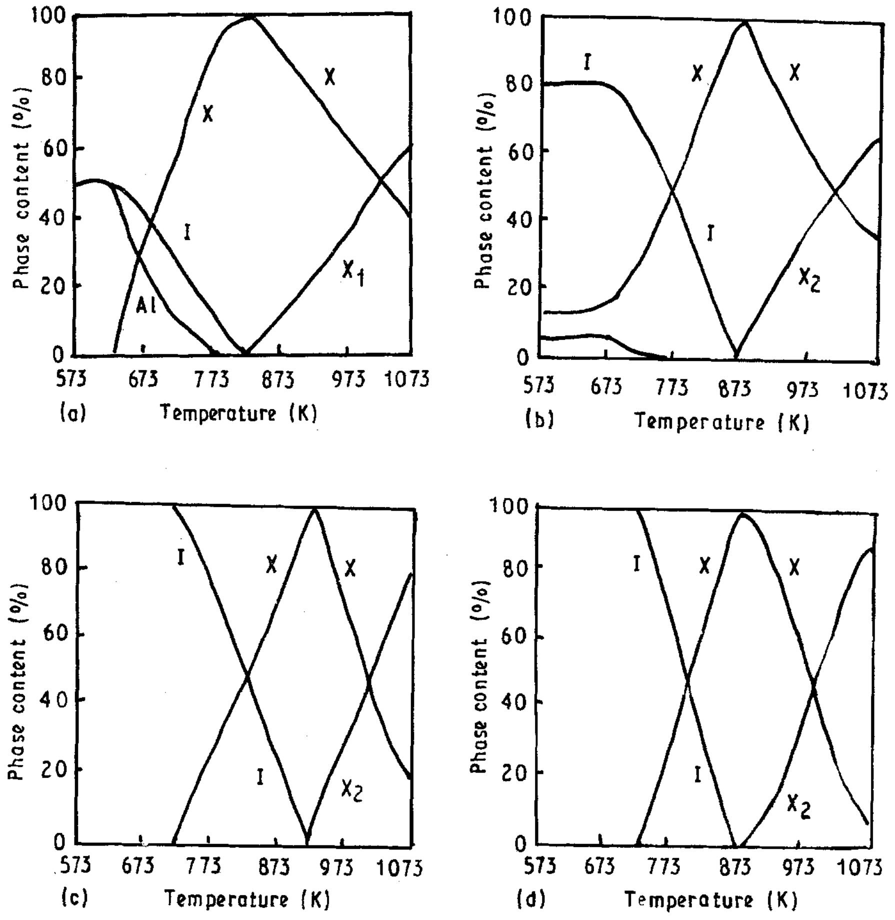
Figure 2 X-ray diffraction patterns of rapidly solidified powder of Al–Cr alloys with different chromium contents (rotating rate of disc and cone is 4200 r. p. m., cooling agent liquid nitrogen).



(a)

(b)

Figure 3 X-ray diffraction patterns of rapidly solidified powders of Al–Cr–Mn alloys with different compositions at different cooling rates: (a) 3200 r.p.m., cooling agent liquid nitrogen; (b) 4200 r.p.m., cooling agent: liquid nitrogen.



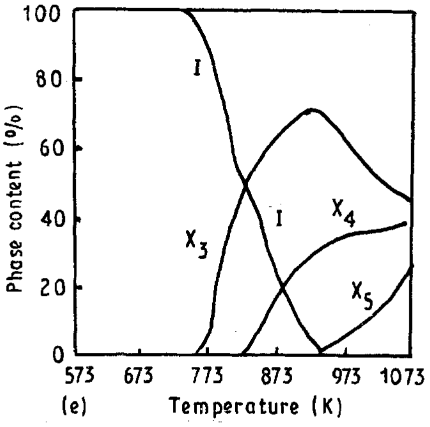


Figure 4 Kinetic phase-transformation diagrams of Al–Cr, Al–Cr–Mn alloy quasicrystalline powders. (a) Al–10Cr, (b) Al–15Cr, (c) Al–18Cr, (d) Al–14Cr–4Mn, (e) Al–4Cr–14Mn.

where q c I is the quasicrystal I phase.

Al–15 at % Cr:

$$\begin{aligned}&\text{RT} - 673\ \text{K})\ \text{fcc Al (small amount)} + \text{qcI} + \\ &\text{X phase} \xrightarrow{673-773\ \text{K}} \text{qcI (decreasing)} + \\ &\text{X phase (increasing)} + \text{fcc Al (decreasing)} \\ &\xrightarrow{773\ \text{K}} \text{qcI} + \text{X phase} \xrightarrow{773-873\ \text{K}} \text{qcI} \\ &\text{(decreasing)} + \text{X phase (increasing)} \\ &\xrightarrow{873\ \text{K}} \text{X phase (maximum)} \xrightarrow{873-1073\ \text{K}} \\ &\text{X phase (decreasing)} + \text{X}_2\ \text{phase (increasing)} \quad (2)\end{aligned}$$

Al–18 at % Cr:

$$\begin{aligned}&(\text{RT} - 723\ \text{K})\ \text{qcI} \xrightarrow{723-923\ \text{K}} \text{qcI (decreasing)} + \\ &\text{X phase (increasing)} \xrightarrow{923\ \text{K}} \text{X phase (maximum)} \\ &\xrightarrow{923-1073\ \text{K}} \text{X phase (decreasing)} + \\ &\text{X}_2\ \text{phase (increasing)} \quad (3)\end{aligned}$$

Al–14 at % Cr–4 at % Mn:

$$\begin{aligned}&(\text{RT} - 723\ \text{K})\ \text{qcI} \xrightarrow{723-873\ \text{K}} \text{qcI (decreasing)} + \\ &\text{X phase (increasing)} \xrightarrow{873\ \text{K}} \text{X phase (maximum)} \\ &\xrightarrow{873-1073\ \text{K}} \text{X phase (decreasing)} + \\ &\text{X}_2\ \text{phase (increasing)} \quad (4)\end{aligned}$$

Al–14 at % Mn–4 at % Cr:

$$\begin{aligned}&(\text{RT} - 763\ \text{K})\ \text{qcI} \xrightarrow{763-823\ \text{K}} \text{qcI (decreasing)} + \\ &\text{X}_3\ \text{phase (increasing)} \xrightarrow{823-923\ \text{K}} \text{qcI (decreasing)} + \\ &\text{X}_3\ \text{phase (maximum)} + \text{X}_4\ \text{phase (increasing)} \\ &\xrightarrow{923\ \text{K}} \text{X}_3\ \text{phase} + \text{X}_4\ \text{phase} \xrightarrow{923-1073\ \text{K}} \\ &\text{X}_3\ \text{phase (decreasing)} + \text{X}_4\ \text{phase (increasing)} + \\ &\text{X}_5\ \text{phase (increasing)} \quad (5)\end{aligned}$$

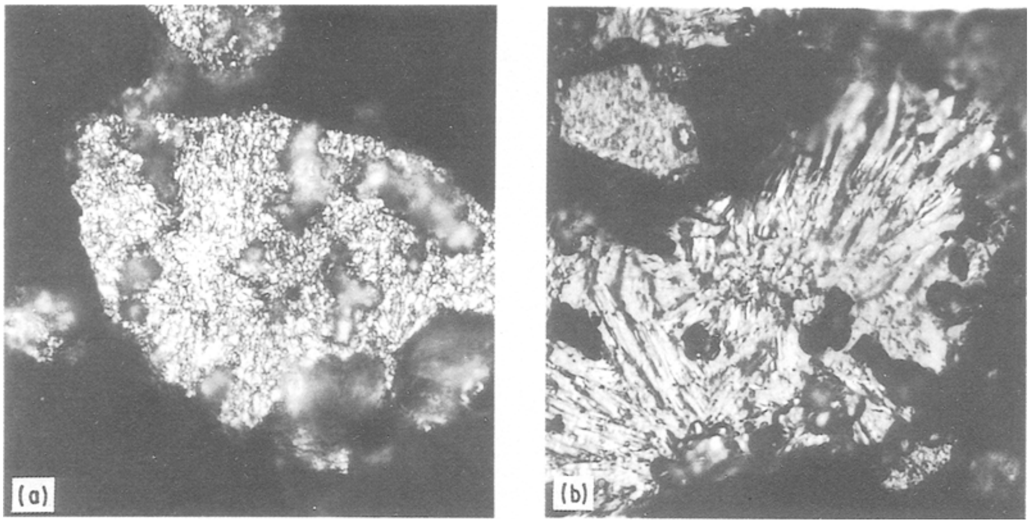


Figure 5 Microstructures of quasicrystalline powders (× 1000): (a) Al–Cr; (b) Al–Cr–Mn.

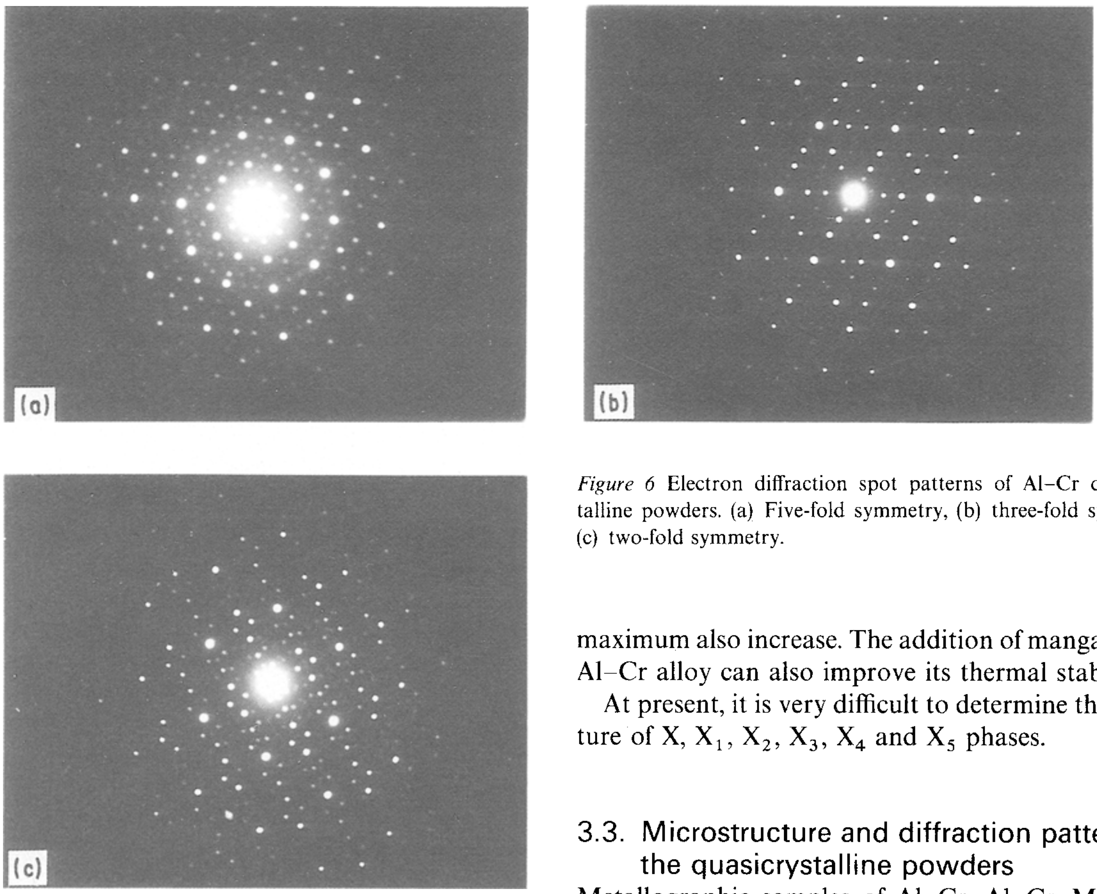


Figure 6 Electron diffraction spot patterns of Al–Cr quasicrystalline powders. (a) Five-fold symmetry, (b) three-fold symmetry, (c) two-fold symmetry.

By means of curve fitting with a quintic orthogonal polynomial as well as the Hook–Jeeves pattern searching method, elevated-temperature X-ray diffraction data were processed [3]. Consequently, the kinetic phase-transformation diagrams of Al–Cr, Al–Cr–Mn quasicrystals were plotted quantitatively (Fig. 4). According to the diagrams, increasing the chromium content in the Al–Cr alloy can improve the thermal stability of the icosahedral phase, i.e. both the original and final transformation temperatures of the I phase increase. Meanwhile, for the metastable phase (X phase) the original temperature from which the amount of X phase begins to increase and the temperature at which the X phase content reaches a maximum also increase. The addition of manganese to Al–Cr alloy can also improve its thermal stability.

At present, it is very difficult to determine the structure of X, X_1, X_2, X_3, X_4 and X_5 phases.

3.3. Microstructure and diffraction patterns of the quasicrystalline powders

Metallographic samples of Al–Cr, Al–Cr–Mn powders were prepared with resin, the sample then were ground, polished and etched with a mixture of acids (Keller's agent). Fig. 5 show the microstructure of these powders.

Fig. 6 shows electron diffraction spot patterns of quasicrystalline powders.

4. Conclusions

1. In Al–Cr, Al–Cr–Mn alloys, the composition and cooling rate dramatically affect the formation of quasicrystalline icosahedral phase. In the present study, nearly fully icosahedral phase quasicrystalline powders can be obtained for $Al_{82}Cr_{18}$, $Al_{82}(Mn, Cr)_{18}$ alloys at a cooling rate of 10^5–10^6 K s^{-1}.

2. The increments of chromium content can improve the thermal stability of the icosahedral phase in the Al–Cr alloy. The addition of manganese in this alloy also improves the thermal stability of the icosahedral phase.

Acknowledgement

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References

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