

MICROSTRUCTURE AND DECOMPOSITION OF RS Al-Fe-Cr-Zr ALLOY^①

Xiao, Yude Li, Shongrui Xie, Yongan Xu, Yiheng

*Department of Materials Science and Engineering,
Central South University of Technology, Changsha 410083*

ABSTRACT

Microstructures and precipitation process of rapidly solidified foil powders of Al-Fe-Cr-Zr have been investigated using TEM, X-ray diffraction, optical microscope and hardness measurement. The results indicate that (1) featureless structure can be obtained easily in Al-Fe-Cr-Zr alloy under rapid solidification condition; (2) $\text{Al}_{13}\text{Cr}_2$, Al_3Zr and $\text{Al}_3(\text{Fe}, \text{Cr})$ phases precipitate during heating; (3) metastable Al_3Zr has noticeable precipitation strengthening effect.

Key words: rapid solidification powder metallurgy heat-resistant aluminum alloy

1 INTRODUCTION

Aluminum alloys developed by means of rapid solidification processing (RSP) for elevated temperature use have been a continuing goal. The interest has stemmed primarily from the need to improve high temperature properties and reduce the weight of components, replace components fabricated from titanium alloy^[1].

At present, the most successfully developed alloy systems are those based on Al-Fe-X^[2] and Al-Cr-Zr^[3]. P/M Al-Fe alloys owe elevated temperature strength to fine metastable Al_3Fe particles formed during heat processing after rapid solidification. However, structural inhomogeneity and high initial hardness create processing difficulties. In contrast, the Al-Cr-Zr alloys are reported to be easier to fabricate and attain strength during or after processing from the precipitation of a coherent metastable Al_3Zr phase. Theoretical consideration suggests that an alloy combining the two systems but with reduced solute content should provide an easily consolidated material with good elevated temperature strength and adequate toughness. The third RS conference of the former USSR reported

that Al-Fe-Cr-Zr(-Mo) alloy at least has more excellent mechanical properties than Al-Fe-V-Si alloy below 350 °C^[4]. Ref. [5] showed that an unidentified phase with iron and chromium rich was discovered in RS Al-Fe-Cr-Zr alloy except for Al_{13}Cr and Al_3Zr dispersions. The objective of this paper is to examine the solidification structure of an Al-Fe-Cr-Zr foil powder produced by a multistage atomization-rapid solidification powder-making technique^[6]. In addition, the decomposition behaviour has been assessed during annealing of the powders.

2 EXPERIMENTAL

The foil powders were produced by a multi-stage atomization rapid solidification powder-making device (cooling rate = $10^5 \sim 10^6$ K/s) and had a melt composition of Al-6.8 Fe-3.75 Cr-1.5 Zr (wt.-%). The ageing powder samples were prepared by choosing the foil powder with 0.1 cm thickness and cutting as square shape ($a = 1$ cm); ageing temperatures were selected as 250, 350, 400 and 500 °C, and hardness was measured by a microhardness meter (load = 3 kg). Phase identification within powder fraction was carried out using

^① Received Apr. 18, 1994; accepted in revised form Aug. 5, 1994

an X650 X-ray diffractometer employing Cu K α radiation at 40 kV and 30 mA. In addition, the unidentified phase was studied using H800 TEM with EPSX. Microstructural characterization was observed by a light microscope. Etch solution used was HF (1.0 mL) + HCl (1.5 mL) + HNO $_3$ (2.5 mL) + H $_2$ O (95.0 mL). Microstructural transformation of foil powder during annealing was assessed by batch heating at 250, 350, 450 and 550 °C.

3 RESULTS AND ANALYSIS

3.1 Outside Character and Construction of Primary Foil Powder

The area and thickness of each foil powder are about 4~6 cm 2 and 1~0.2 cm.

Preliminary details of the as-received powder microstructures were presented in ref. [7]. The primary foil powder as ordinary rapidly solidified powder exhibited either cellular (B-zone) or structurally featureless morphologies (A-zone), but most commonly a mixture of both, see Fig. 1. It is possible to characterize the microstructure in relation to the foil powder's thickness. B-zone will be larger in thicker powder, sometimes with coarse second phase. A-zone will be increased with reduction of thickness, and only A-zone in very thin foil powder. It shows that complete supersaturated solution can be obtained under very rapid solidification, which is noticeable according to X-ray diffraction. Hence it is apparent that the Al-Fe-Cr-Zr alloy is

more susceptible to partitionless solidification and easier to obtain complete supersaturated solution.

3.2 Identification of Unidentified Phase

X-ray analysis indicates that no equilibrium phases are formed in the foil powder except thick powder during rapid solidification. With elevation of temperature and increasing of ageing time, Al $_3$ Zr, Al $_{13}$ Cr $_2$ and an unidentified phase containing rich iron and chromium will exist in the powder. Through TEM and EDXS this unidentified phase could be identified to be Al $_3$ (Fe, Cr) (Fig. 2), which is in fact formed by partial substitution of chromium to iron of Al $_3$ Fe intermetallic compound.

At elevated temperature, metastable θ -Al $_3$ Fe

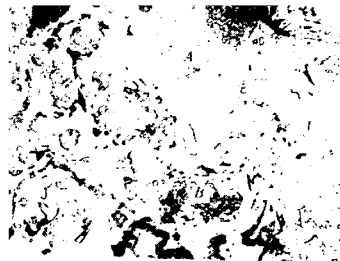


Fig. 1 Structure of rapidly solidified powder

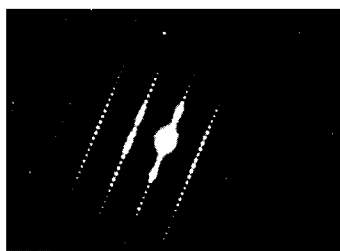
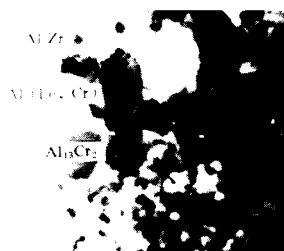


Fig. 2 TEM showing that unidentified phase is Al $_3$ (Fe, Cr)
(a)—phase morphology; (b)—diffraction pattern of Al $_3$ (Fe, Cr)

transforms into the monoclinic equilibrium phase $\text{Al}_{13}\text{Fe}_4$.

3.3 X-ray Diffraction of Ageing Samples

From the change of X-ray diffraction peak, the phase transformation in the samples can be found out, see Fig. 3.

When kept at 250 °C for 16 h, except few strong peaks correspond to $\text{Al}_{13}\text{Cr}_2$, there is almost no peak of other second phase, which proves that metastable supersaturated solution has comparative stability and $\text{Al}_{13}\text{Cr}_2$ is easier to precipitate from aluminum matrix. After annealed for 32 h, some

stronger diffraction peaks of stable DO_{23} Al_3Zr appear, which shows that the thermo-stability of metastable phase Al_3Zr is excellent. Metastable LI_2 Al_3Zr has *fcc* structure ($a = 4.051 \sim 4.071 \text{ \AA}$), which is very similar to aluminum matrix (*fcc*, $a = 4.049 \text{ \AA}$). Hence diffraction peak of LI_2 Al_3Zr is neglected in those of aluminum and difficult to be observed^[8]. Metastable $\text{Al}_3(\text{Fe}, \text{Cr})$ needs longer time for pregnancy and for heating before appearing, which might be due to low diffusion coefficient of iron atom.

At 350 °C, precipitation of all second phases get ahead, especially $\text{Al}_3(\text{Fe}, \text{Cr})$, which used to precipitate after stabilization of metastable Al_3Zr at

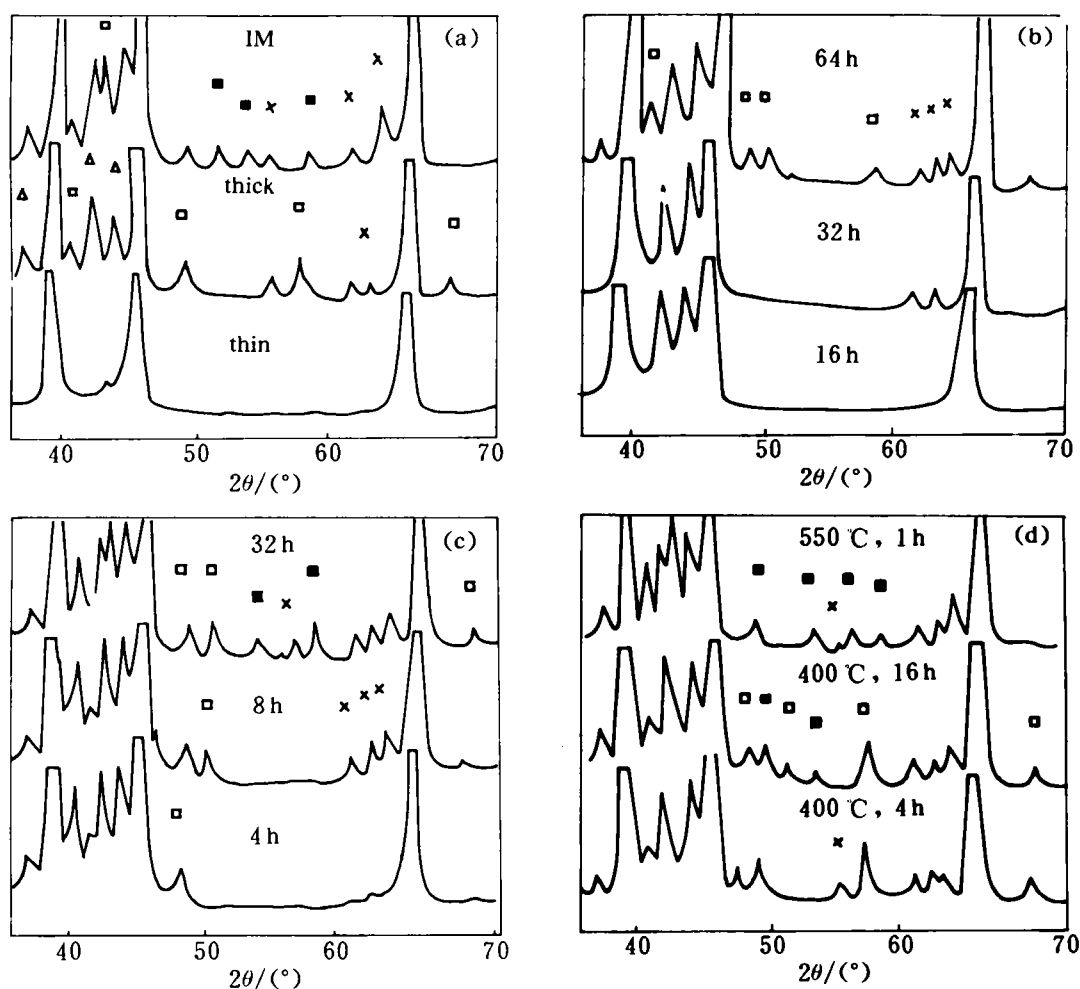


Fig. 3 X-ray diffraction diagrams of Al-Fe-Cr-Zr alloy

(a)—primary samples of foil powder and ingot; (b)—ageing sample at 250 °C; (c)—that at 350 °C; d—those at 400, 550 °C; \triangle — $\text{Al}_{13}\text{Cr}_2$; \times — Al_3Zr ; \square — $\text{Al}_3(\text{Fe}, \text{Cr})$; \blacksquare — $\text{Al}_{13}(\text{Fe}, \text{Cr})_4$

250 °C, form ahead to stable Al_3Zr , which explains that metastable Al_3Zr has excellent stability.

At 400 °C annealing for short time, all of $\text{Al}_{13}\text{Cr}_2$, stable Al_3Zr and $\text{Al}_3(\text{Fe}, \text{Cr})$ appear noticeably, and after longer time ageing $\text{Al}_3(\text{Fe}, \text{Cr})$ phase reduces while equilibrium phase $\text{Al}_{13}\text{Fe}_4$ forms, which may be due to the fact that $\text{Al}_3(\text{Fe}, \text{Cr})$ phase has transformed to $\text{Al}_{13}\text{Fe}_4$ phase at high temperature.

During short time at higher temperature, such as 550 °C, $\text{Al}_{13}\text{Cr}_2$, stable Al_3Zr , $\text{Al}_3(\text{Fe}, \text{Cr})$ and $\text{Al}_{13}\text{Fe}_4$ form rapidly in the ageing sample, which is almost the same as ingot sample seen from the analysis of X-ray diffraction.

3.4 Ageing Curves of Foil Powders

Fig. 4 is the curves of hardness changing with ageing time at 250, 350, 400 and 500 °C respectively. The peak of ageing hardening was noticeable on the curves of 250, 350 and 400 °C, among which there are two peaks on that of 350 °C, and with elevating temperature, the peak disappears and is not evident.

During the starting stage, especially at lower temperature, the hardness change is unnoticeable. During last period, the hardness remains nearly stable at lower temperature, but when ageing at 400 °C the hardness reduces noticeably, which could be connected to the coarsening of the second

phase. However, at 500 °C, the hardness increases lightly on the contrary, which might be due to powder sintering.

4 DISCUSSION

The solidification process of Al-Fe-Cr-Zr powder can be explained through powder size because various dimension signifies different cooling rate^[5]. With powder dimension reducing, heat conductive coefficient increases, while maximum recalescent temperature becomes lower. A-zone is easier to form, namely, the microstructure of alloy develops from fine segregated structure to cell and then featureless morphology as cooling rate increases.

The foil powder produced by rapid solidification is metastable supersaturated solid solution (A-zone). During heating, second phase particles precipitate, A-zone contracts while B-zone enlarges. $\text{Al}_{13}\text{Cr}_2$ has priority to precipitate because of larger diffusive coefficient of chromium atom in aluminum, while $\text{Al}_3(\text{Fe}, \text{Cr})$ precipitation needs longer pregnancy and falls behind to stabilization of metastable Al_3Zr at low temperature. But at elevated temperature, $\text{Al}_3(\text{Fe}, \text{Cr})$ precipitates earlier than stabilization of Al_3Zr . At much higher temperature, metastable Al_3Zr and $\theta\text{-Al}_3\text{Fe}$ phases transform to $\text{DO}_{23}\text{Al}_3\text{Zr}$ and $\theta\text{-Al}_{13}\text{Fe}_4$ equilibrium phases while particles of second phase coarsen. During ageing, change of alloy hardness results commonly from two effects of reduction of solution strengthening caused by decreasing solution content and precipitation strengthening caused by precipitation of second phases. In the RS Al-Fe-Cr-Zr alloy, metastable Al_3Zr is coherent with aluminum matrix and has noticeable dispersion strengthening effect^[9]. This leads to the hardness peak of 250 °C ageing curve, while precipitation of $\text{Al}_{13}\text{Cr}_2$ and $\text{Al}_3(\text{Fe}, \text{Cr})$ decrease the hardness. At the initial stage of ageing, although a little $\text{Al}_{13}\text{Cr}_2$ phase precipitates, it has less effect on supersaturated solid solution state of the alloy and the sample is still supersaturated, the hardness of the materials is almost constant. When metastable Al_3Zr precipitates and its precipitation hardening effect plays a leading role, the alloy is obviously hardened. With

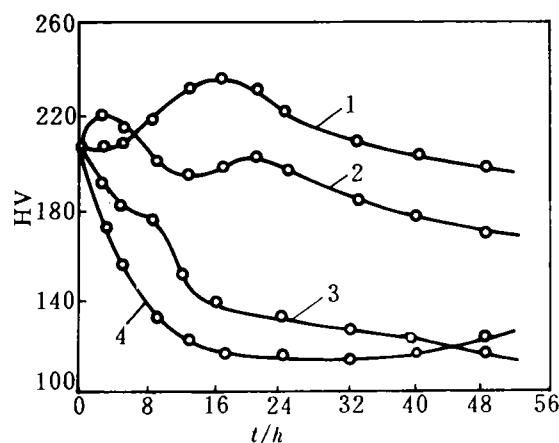


Fig. 4 Ageing curves of thin foil powders at different temperatures

1—250 °C ; 2—350 °C ; 3—400 °C ; 4—500 °C

$\text{Al}_{13}\text{Cr}_2$ and Al_3Fe phase precipitation one after another, ageing hardening caused by precipitation of metastable Al_3Zr is offsetted, ageing peak of the curves is decreased, and under certain condition, there might be two peaks on ageing curve. Ageing at high temperature, because a large amount of second phases precipitate for short time and metastable Al_3Zr phase transforms to equilibrium phase rapidly, ageing hardening effects become more unnoticeable. Therefore, for 400 °C ageing curve, the hardness decreases on the whole except for a cushion during dropping; but for 500 °C ageing curve, no ageing hardening effect is observed, hardness of the alloy decreases significantly for short time. In addition, ageing at high temperature for long time, both stabilization of metastable phases and ripening of second phases are important for the hardness to decrease further.

5 CONCLUSIONS

(1) The Al-Fe-Cr-Zr alloy is more susceptible to partitionless solidification and complete A-zone structure can be obtained under $10^5 \sim 10^6$ K/s cooling rate.

(2) During supersaturated solid solution precipitation, metastable Al_3Zr , $\text{Al}_{13}\text{Cr}_2$ and $\text{Al}_3(\text{Fe}, \text{Cr})$ phases formed in the alloy. At low temperature, precipitation of $\text{Al}_3(\text{Fe}, \text{Cr})$ phase is later

than stabilization of metastable Al_3Zr . But at high temperature metastable Al_3Zr phase shows high stability. At higher temperature, stable Al_3Zr and $\text{Al}_{13}\text{Fe}_4$ phases can be found.

(3) Metastable Al_3Zr phase has noticeable precipitation hardening effect, and in fact hardness change of the alloy is caused commonly by two effect: dispersion hardening of metastable Al_3Zr and softening caused by the precipitation of $\text{Al}_{13}\text{Cr}_2$ and $\text{Al}_3(\text{Fe}, \text{Cr})$ phases.

REFERENCES

- 1 Chellman, D J *et al.* Metal Powder Report, 1988, 43 (10): 672.
- 2 Boettinger, W J; Bendersky, L *et al.* Met Trans, 1986, 17A: 781.
- 3 Allborned, H; Merg, D *et al.* Aluminium, 1971, (47):
- 4 Froes, F H. In: The Third All-Union Soviet States Conference on Rapidly Solidified Alloys. JOM, 1991.
- 5 Marshall, C J. Journal of Materials Science, 1987, 22: 3581—3588.
- 6 Chen, Zhenghua *et al.* CN 90106130. 1.
- 7 Marshall, G J. In: Sheppard, T(ed), Aluminium Technology "86". London: Institute of Metals, 1986, 676.
- 8 Michot, Gerard; Champier, Georges. Physical Metallurgy of P/ M Aluminium Alloys. Beijing: ICCAA2, 1990.
- 9 Marshall, G J; Hughes I R *et al.* Mater Sci Technol, 1986, 2: 394.