THE CONSOLIDATION OF QUASICRYSTALLINE POWDER UNDER SUPERHIGH PRESSURE①

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ABSTRACT

Al-based quasicrystalline powders were consolidated by a new technique. This paper describes the consolidation process and the properties of bulk quasicrystalline materials. The bulk, dense, full quasicrystalline samples were obtained by means of consolidating quasicrystalline powders at superhigh pressure below their crystallizing temperature or at low temperture. At superhigh pressure, the thermal stability of the quasicrystalline powders increased, while the quasicrystalline powders containing some crystalline phases were purified, that the crystalline phases disappeared and the amount of quasicrystalling phases increased. Besides, the consolidated materials possessed high strength and were free from flaws and Mach holes caused by shock waves; therefore, this method is better than the explosion consolidation technique.

Key words: powder metallurgy, compaction / rapid solidification, quasicrystals

1 INTRODUCTION

One important way through which quasicrystalline materials can be applied to industry is to consolidate quasicrystalline powders produced with rapid solidification devices into bulk samples. Because of their hardness and brittleness, it is difficult to compact and consolidate quasicrystalline powders. After a careful study on the explosion compaction of quasicrystalline powders, we investigated a new method— the superhigh static pressure consolidation technique, which means to consolidate quasicrystalline powders at a very high pressure (e. g. $>$5 GPa) at the temperature below their crystallization (T_c). This technique is obviously superior to explosion compaction; namely, samples prepared with this method are high in strength, flawless, and free from pores and holes; they preserve fully the icosahedral structures of their raw powders.

2 EXPERIMENTAL

In this study, the rapid solidification device developed in our lab was used to produce quasicrystalline powder[1]. The average particle size of produced powder is 15–20μm. The powders were consolidated at a pressure of 5–7 GPa with a hexahedral apparatus usually used to synthesize diamond. In this process, powder compacts were heated with a graphite tube to the resistance of the compact itself. As shown in Fig.1, the thermocuple was put

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directly inside the powder compact. The pyrophyllite block was first pressured at roomte mperature to 5–7 GPa; then the compact was heated to required temperature within 30 seconds. With such a process, the oxidation of the powder during the heating in air could be greatly depressed. The consolidated sample was cooled to room tem- perature at high pressure in 2–3 min and after the pressure was unloaded, the bulk quasicrystalline sample was picked up.

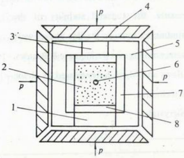

Fig.1 Sketch of superhigh pressure consolidation apparatus

1–electrode; 2–powder;
3–electrode; 4–anvil;
5–pyrophyllite block; 6–thermocouple;
7–graphite tube; 8–graphite disc

3 RESULTS AND DISCUSSION

3.1 *Phase Structures and Diff raction Patterns of Consolidated Qusicrystalline Samples*

Full quasicrystalline powders of $Al_{77.5}Mn_{22.5}$ and $Al_{65}Cu_{20}TM_{15}$(TM = Fe,Cr,Mn) were used. They were consolidated at a pressure of 5–7 GPa and at a temperature below T_c for 1–10 min. The prepared samples maintain the same structure of their raw powders— the icosahedral structure. Fig.2 demonstrates the electron diffraction patterns of the bulk quasicrystalline samples (with H–800 TEM). Fig.3 shows the morphology of the bulk quasicrystalline sample (with H737 SEM).

Fig.4 is an X–ray diffraction (XRD) spectra of consolidated $Al_{77.5}Mn_{22.5}$ samples. When consolidated at lower temperature and at higher pressure, the obtained samples fully maintained the diffraction characteristics of quasicrystals; when at about crystallizing temperature and at a lower pressure, or above crystallizing temperature (e.g. at 813 K) at a higher pressure for a short time, the consolidated materials basically retained the diffraction spectrum characteristics of the icosahedral phase except for some broadening in the width of the spectrum peaks. This broadening

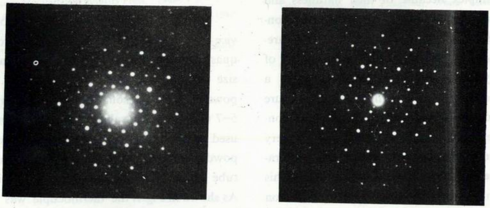

Fig.2 5–3–2 Fold symmetry of electron diffraction patterns of $Al_{77.5}Mn_{22.5}$ bulk sample

effect is similar to that occurred in the explosion compaction of quasicrystalline powder[2]. This broadening effect was probably not only the results of internal stress, but it may also proceed from the supposed trace crystalline phases formed during consoli- dation. Therefore, these facts indicated that the thermal stability increased when consolidating at superhigh pressure. But, if the consolidating temperature is high enough, (e. g. 893 K)the crystallizing would occur even at a high pressure.

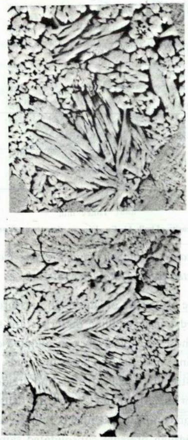

Fig.3 Morphology of bulk $Al_{77.5}Mn_{22.5}$ sample

Fig. 5 shows the XRD spectra of consolidated $Al_{65}Cu_{20}TM_{15}$ quasicrystalline samples. Because both $Al_{65}Cu_{20}Fe_{15}$ and $Al_{65}Cu_{20}Cr_{15}$

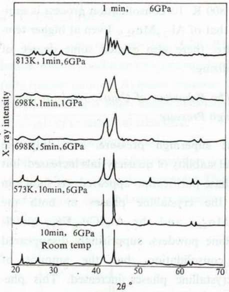

Fig.4 XRD spectra of consolidated $Al_{77.5}Mn_{22.5}$ samples

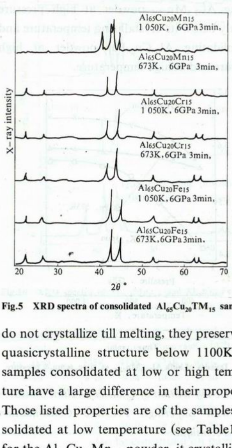

Fig.5 XRD spectra of consolidated $Al_{65}Cu_{20}TM_{15}$ samples

do not crystallize till melting, they preserve the quasicrystalline structure below 1100K, but samples consolidated at low or high temperature have a large difference in their properties. Those listed properties are of the samples consolidated at low temperature (see Table1). As for the $Al_{65}Cu_{20}Mn_{15}$ powder, it crystallizes at about 800 K. Its consolidation process is similar to that of $Al_{77.5}Mn_{22.5}$. Even at higher temperature, there also existed some degree of crystallizing.

3.2 *The Behaviour of Quasicrystals at Superhigh Pressure*

At superhigh pressure, not only the thermal stability of quasicrystals increased, but also phase purification appeared. As shown in Fig.6 the crystalline phases in both the $Al_{77.5}Mn_{22.5}$ and the $Al_{65}Cu_{20}Fe_{15}$ quasicrystalline powders, supprisingly disappeared after consolidation, but the amount of quasicrystalline phases increased. This phenomenon was very obvious when consolidating $Al_{77.5}Mn_{22.5}$ powder at high pressure slightly below its crystallizing temperature and consolidating $Al_{65}Cu_{20}Fe_{15}$ powder at high pressure and at high temperature.

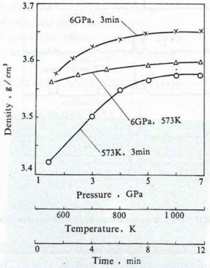

Fig.6 Density of bulk $Al_{77.5}Mn_{22.5}$ vs time, temp.& pressure

3.3 *Densification of Quasicrystalline Powders with Superhigh Pressure Consolidation Technique*

Densities of quasicrystalline samples depend on consolidating parameters. Fig.7 shows correlation between density and pressure, temperature and time for $Al_{77.5}Mn_{22.5}$, $Al_{65}Cu_{20}Fe_{15}$, $Al_{65}Cu_{20}Cr_{15}$ and $Al_{65}Cu_{20}Mn_{15}$ alloys respectively. Optical microscope and SEM observations show that, in general, samples consolidated at P > 5 GPa, t > 5 min, T = 673 K are dense and pore-free.

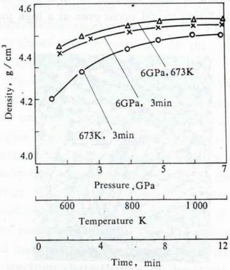

Fig.7 Density of bulk $Al_{65}Cu_{20}Fe_{15}$ vs time, temp & pressure

3.4 *Properties of Superhigh Pressure Consolidated Quasicrystalline Materials*

The properties of superhigh pressure consolidated quasicrystalline materials are superior to those of explosively consolidated quasicrystalline materials. These materials have higher strength, less brittleness and are denser, (e. g. for the $Al_{77.5}Mn_{22.5}$ alloy the density of explosion compacted sample is 3.43 g/cm³; as for superhigh pressure consolidated sample it is 3.62 g/cm³); besides, they are flawless and free from Mach holes caused by shock waves. In particular, by using this technique one can produce quasicrystalline materials in large

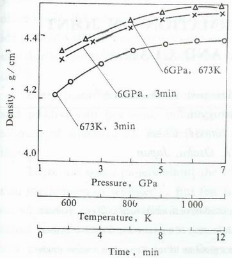

Fig.8 Density of bulk $Al_{65}Cu_{20}Mn_{15}$ vs time, temp. & pressure

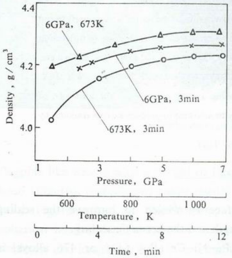

Fig.9 Density of bulk $Al_{65}Cu_{20}Cr_{15}$ vs time, temp.& pressure

quantity, which provides a new way for industrial production of quasi- crystalline materials.

4 CONCLUSIONS

1 At very high pressure (5–7 GPa) and below their crystallizing temperature for 5 min, fully quasicrystalline Al-based powders can be consolidated into bulk, dense quasicrystalline materials of icosahedral structure.

2 At superhigh pressure, the thermal stability of quasicrystalline powders increased and the phase purification occurred. The superhigh pressure consolidation is superior to explosion compaction.

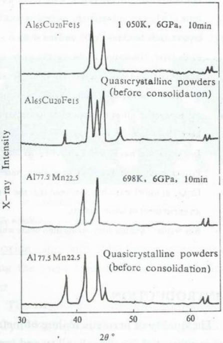

Fig.10 XRD spectra of $Al_{77.5}Mn_{22.5}$ and $Al_{65}Cu_{20}Fe_{15}$ powders with crystal phase & consolidated materials

Table 1 Test results of the properties of quasicrystal powders

matenal	deasity (g / cm³)	hardness HK(kg / mm²)	resistivity (μ · Ωm)	linear expansion rate(× 10⁻⁶) 373K	473K	573K	673K	773K
$Al_{77.5}Mn_{22.5}$	3.62	780–800	8–10	8.48	9.24	10.40	11.80	11.20
$Al_{65}Cu_{20}Fe_{22.5}$	4.50	800–850	20–22	15.84	24.54	25.84	29.42	31.60
$Al_{65}Cu_{20}Mn_{15}$	4.30	750–800	16–18	16.41	24.00	24.14	25.96	26.02
$Al_{65}Cu_{20}Mn_{15}$	4.40	750–780	18–20	17.21	26.26	28.54	28.80	28.94

(to be continued on page 78)