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Investigation of $Al_{62.5}Cu_{25}Fe_{12.5}$ Quasicrystalline Phase Formation by Rapid Solidification Followed by Mechanical Milling

استقصاء تكوين الطور شبه البللورى لسبيكة $Al_{62.5}Cu_{25}Fe_{12.5}$ بطريقة السبك الميكانيكى الذى يلى التجمّد السريع

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ملخص البحث

تمت دراسة أحتمال تكون الطور شبه البللورى لسبيكة $Al_{62.5}Cu_{25}Fe_{12.5}$ عن طريق أستخدام طريقة التجمد السريع بإستخدام تقنية التوام الأسطوانى. لقد تم أستخدام تقنيات حيود الأشعة السينية، المعالجة الحرارية،و قياس المقاومة الكهربية لدراسة خصائص العينات التى حصلنا عليها. دراسة تأثير الطحن على العينات التى حصلنا بتقنية التوام الأسطوانى قد تمت دراستها أيضاً.

بالنسبة للعينات التى بردت فجائياً فقط لقد تم الحصول على الطور شبه البللورى بنسبه عالية، بالأضافة الى طورين آخرين هما الطور Al_2Cu والطور Al_7Cu_2Fe (ω-phase) أختبار اضافة الطحن للعينات المتكونة بالتبريد الفجائى لم يعطى طور أحادى للطور شبه البللورى، و لكن الطحن الميكانيكى ثبت الطور الذى حصلنا عليه فعلياً.

ABSTRACT

The formation of the quasicrystalline phase $Al_{62.5}Cu_{25}Fe_{12.5}$, by twin rolls rapid solidification technique has been investigated. The obtained phases are cauterized by XRD, Differential Thermal Analysis and electrical resistivity measurements. The effect of milling on the sample produced by rapid solidification is also examined.

In the as quenched sample, a quasicrystalline (i-phase) is present in a relative high volume fraction, in addition to two other phases that are most likely to be Al_2Cu phase and Al_7Cu_2Fe phase (ω-phase). The milled samples does not give a single quasicrystalline phase, but the milling tends to stabilize the pre existed i-phase.

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1. INTRODUCTION

Since the production of the first quasicrystalline phase AlMn in 1984 [1] and then the production of the first stable and perfect quasicrstallline phase with high structure quality in 1987 in the rapidly solidified AlCuFe alloy [2], the quasicrystals have been the subject of intense studies. The reason of this great interest of quasicrystals lies of their exceptional structure, the presence of orientational five fold structure and a perfect long range order but with no translational periodicity [3,4]. Moreover, those type of structural materials have attractive properties such as, very low electrical and thermal conductivity, [5,6] high hardness accompanied by high brittleness at room temperature [7,8] low surface energy, low coefficient of friction and high resistance to corrosion [9,10].

The rapid solidification represents, until now, one of the most important methods for producing the quasicrystalline phase. Earlier studies concerning the production of quasicrystalline phase [11,12] indicated that, it is possible to obtain a pure quasersta!lline phase, i-phase, in AlCuFe by rapid solidification followed by annealing at 800° C for nearly one hour. The as quenched sample contains a small amount of cubic β-AlFe phase and a considerable amount of structural defects., which are both eliminated by annealing treatment [13].

In a more recent studies have reported that [14-16] the production of pure i-phase obtained by rapid solidification is very sensitive to many parameters of production, such as the nominal composition, the technique used to achieve the rapid solidification, the cooling rate and the presence of inert gas atmosphere.

Another important method to produce the quasicrystalline phase is the mechanical alloying, MA, [17,18]. It was reported that, the MA tends to enhance the formation of the i-phase [18,19] for certain ternary alloyed systems.

The present work is an attempt to examine the possibility of formation of the i-phase of the composition $Al_{62.5}Cu_{25}Fe_{12.5}$, which lies in the region of the perfect i-phase in the ternary phase diagram [20], under certain production parameters. Also, the effect of milling on the produced sample by rapid solidification method is examined.

2. EXPERIMENTAL PROCEDURE

A sample of nominal composition $Al_{62.5}Cu_{25}Fe_{12.5}$ was prepared by rabid solidification of the melt. Commercial iron sheets, electrical copper sheets, and an $AlCu_{25}$ master alloy of purity 99.9% were used. The master alloy and the elements were melted together in an electrical resistance furnace, of type Lenton furnace with maximum temperature 1500° C. The master alloy and the copper have been melted together firstly at nearly 1100° C, then the iron was added at a temperature of 1250° C.

The melt was rapidly solidified by projecting it into twin rolls system which consists of two pure copper cylinders 120 mm long, 70 mm outer diameter, rotating at opposite directions with rotational speed 1400 rpm. The separation between the two rolls is in the range of 50-100 μm. In this way thin ribbons of dimensions ≈ 0.25 x 4 x 15 mm were obtained.

The thermal stability of as quenched sample was examined by differential thermal analysis (DTA). Perkin-Elmer DTA, computer controlled. The sample was heated at constant heating rate 15° C/s in ambient air, static air, as furnace atmosphere.

The as quenched samples are then annealed at temperatures 680° C and 800° C for one hour, and then rapidly cooled in air. The thermal stability of the annealed sample are also examined by DTA.

The produced phases in all samples are characterized by XRD, using Siemens D5000 computer controlled diffractometer with nickel filter copper K_α radiation, $\lambda = 0.15406$ nm, The diffractometer was operated with step time one second and step size 0.05 degree as a continuous mode. The X-ray tube operates at accelerating voltage 30 kV and current 30 mA.

The electrical resistivity of the as quenched and annealed samples, and its dependence with temperature was measured by the four-probe method. On the other hand, to study the effect of mechanical milling on the sample prepared by rapid solidification, a 15 g of the sample prepared by rapid solidification followed by annealing at 800° C for one hour was grinded and placed in attritor with 150 g balls of different sizes. The milling process was performed for 10 h, 15 h, and 20 h. The milling speed was 500 rpm and it took place under Argon atmosphere in order to prevent any possible oxidation during milling.

3. Results AND DISCUSSIONS

3.1 The Sample Prepared by Rapid Solidification

3.1.1 The as quenched sample

The as quenched sample, diffraction pattern is presented in Fig. (1a). From this pattern, it can be seen that a quasicrystalline (i-phase) is presented in a relative high volume fraction, in addition to two other phases that are most likely to be Al_2Cu phase and Al_7Cu_2Fe (ω-phase).

The fact that under present rapid solidification that more than one phase appears, indicate that the quasi-phase formation is processing dependent. Furthermore, since two other phases took place in addition to i-phase might well indicate that the three phases are formed during the rapid cooling stages. Since melting temperature is a similar to many other rapid subject attempts.

The DTA analysis of the as-quenched, shown in Fig. (2), structures indicated only a single exothermic peak at 670° C. In order to follow the various phases thermal stability, annealing were carried at two temperatures 680° C and 800°C. The electrical resistivity of the as quenched sample is measured at room temperature giving a resistivity value of 625 μΩ cm, which is meanly related to the presence of the icosahedral AlCuFe phase, of high resistivity value, usually 1000 μΩ cm, [21] and to the two other metallic conducting phases with a relative small values of resistivity.

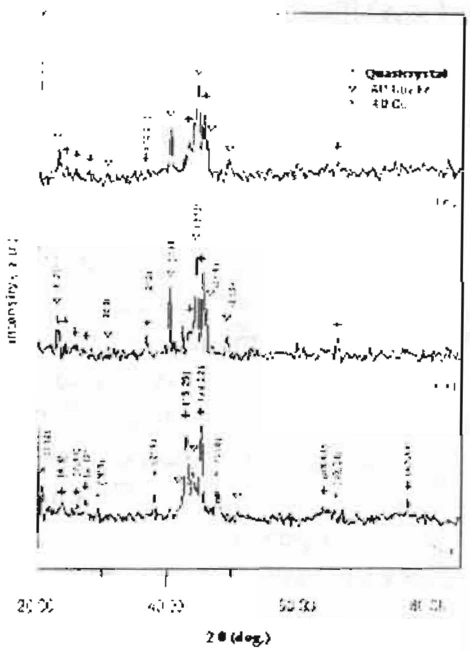

Fig. 1: X ray diffraction pattern of the sample a) As quenched, b) Annealed at 680° C for 1 h, c) Annealed at 800° C for 1 h

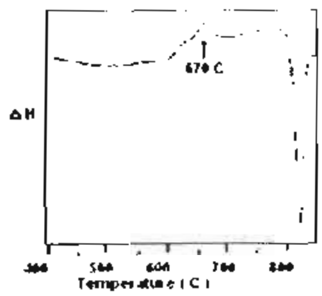

Fig. 2: DTA curve of the as quenched sample

The temperature dependence of resistivity from 20° C up to room temperature is shown in Fig (3), the electric resistivity is observed to increases linearly up to room temperature which is the usual behavior of the ordinary metals. By comparing the measured resistivity ratio ($\rho_{20} / \rho_{295} = 0.42$) for the as quenched sample, with that of the ordinary metals at this range of temperature, 0.1 or less, and also with that of the quasicrystaline $Al_{62.5}Cu_{25}Fe_{12.5}$ phase, in the order of 2.0, the present different value could well be explained again due to the coexistence of the i-phase with metallic phases.

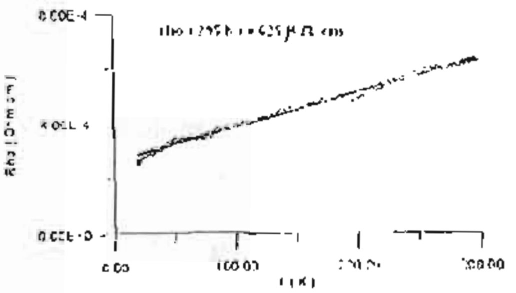

Fig. 3: Variation of electrical resistivity with temperature for the as quenched sample

3.1.2 The sample annealed at 680° C

The XRD pattern of the sample annealed at 680° C for one hour is shown in Fig. (1. b). Following such, it is observed that after this treatment, the Al_2Cu phase disappeared completely, and the amount of the Al_7Cu_2Fe phase increased to large extent. This results indicate the unstability natural at this phase by which when it is thermally added it will dissolve and transform rather to the more stable Al_7Cu_2Fe phase.

The DTA curve of the sample after annealing at 680° C shown in Fig. (4) indicates that, there is no further possible phase transformation to be expected. Measurements of the electrical resisitivity at room temperature of the sample annealed at 680° C gives the value of 460 μΩ cm. This value is less than the value obtained for the as-quenched sample, which could be due to the dissolution of the Al_2Cu phase and the increasing of the volume fraction of the ω phase.

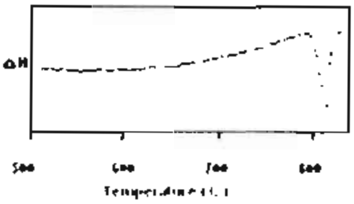

Fig. 4: DTA curve of the sample annealed at 680° C for 1h

3.1.3 The sample annealed at 800° C

The X-ray diffraction pattern and the DTA curves of the sample annealed at 800° C are shown in Fig. (1. c) and Fig (5) respectively. The thermograph indicated are in similar trend to one obtained after annealing at 680° C, which proves that, annealing at higher temperatures did not cause any further transformation. This result fits with the result of the electric resisitivity measurements at room temperature, 470 μΩ cm. Further phase transformations did not expected is proved from the temperature dependence with resisitivity of the sample annealed at 800° C. We found the resistivity increases as the temperature increases, which is normal metallic behavior Fig. (6).

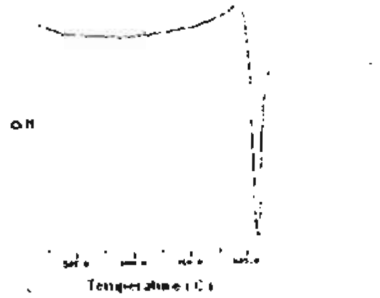

Fig. 5: DTA curve of the sample annealed at 800° C

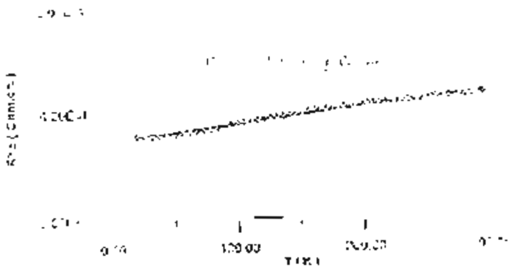

Fig. 6: Variation of electrical resistivity with temperature for the sample annealed at 800° C

3.2 The Sample Prepared by Rapid Solidification Followed by Mechanical Milling

To investigate the effect of various milling times on the sample produced by rapid solidification after the thermally treated at 800° C. The XRD patterns of the various milled samples are shown in Fig. (7). The patterns indicate that, with the increase of the milling time, the peaks of the ω phase (Al_7Cu_2Fe) decrease in its intensity and also in their number, and disappear completely after 15 h of milling. On the other hand, with increasing the milling time, the icosahedral phase is still presented in an appreciable volume fraction, in a way that, it may be thought that the process of milling tends to enhance the i-phase formation. It is also to be noted that, the peaks of the i-phase are broadened with the increase of the milling time, which is expected due to the mechanical straining present during the process of milling. A new undefined phase began to appear after 10 h of milling, and this phase continue to increase with further milling but there is not enough lines to be indexed.

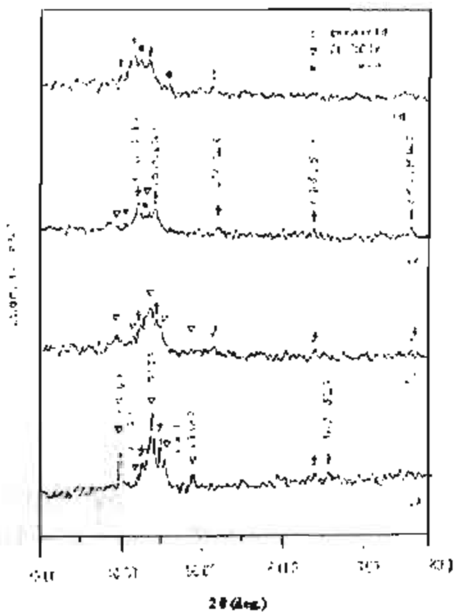

Fig. 7: X-ray diffraction pattern of sample prepared by rapid solidification followed by annealing. a) before milling, b) after 5 h milling, c) after 10 h milling, d) after 15 h milling

4. CONCLUSION

It is possible to obtain a quasicrystaline phase (i-phase) by rapid solidification technique under the present conditions, but not as a single phase even after annealing.. Two phases were obtained instead after annealing (i-phase + ω phase). Such deviation could well be related to slower rate of cooling lead to certain composition separation.

This deviation can be due to some material losses during the process of alloy melting. Thermal analysis of the annealed structure prove its stability up to melting temperature. Addition milling to rapid solidification structure did not lead to massive transformation to i-phase.

5. References

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