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Intermetallic Formation In Mechanically Alloyed AlMnsi Powders.

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تكون الطور بين الفلزى خلال السبك الميكانيكى لمسحوق الألمونيوم والمنجنيز والسليكون .

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ملخص البحث :-

تمت دراسة تكون الطور بين الفلزى وكذا الطور غير المتبلر لسبيكة الألمونيوم منجنيز سيلكون بواسطة طريقة السبك الميكانيكي مبتدئاً بمسحوق متبلر لهذه العناصر وتم تتبع عملية السبك بواسطة الأشعة السينية والمحلل التفاضلي السعرى وكذا البنية المجهرية بواسطة المجهر الإلكتروني المساح وقد وجد إن الطور بين الفلزى Mn_{12} Si_7 Al_5 المتبلر يتكون مباشرة أثناء عملية الطحن لمساحيق العناصر بالتفاعل الإنتشارى الداخلى فى الحالة الجامدة بينما يتكون طور غير متبلر جزئياً بعد ١٣٥ ساعة من الطحن وقد تمت مقارنة هذه النتائج بالنتائج والبيانات التى تم الحصول عليها عن طريق التبريد فائق السرعة لهذا النوع من السبائك .

Abstract

The formation of crystalline intermetallic and amorphous phases by mechanical alloying from crystalline elemental powders has been investigated for AlMnSi alloy system. The alloying process has been monitored by X-ray diffraction (XRD), and differential scanning colorimetry (DSC). The microstructure has been characterized by scanning electron microscopy (SEM).

$Mn_{12}Si_7Al_5$ crystalline intermetallce phase forms directly form the starting materials during milling by a solid state interdiffusion reaction, while a partial amorphous phase is obtained after 135 hours milling. The results are compared with data for rapid solidification of the alloy system.

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Introduction

The structure changes observed during mechanical alloying and the reaction mechanism in the powder during milling process is still controversy. The large number of structural defects produced gives rise to a solid-state type reaction which has been proposed to explain the structural changes during mechanical alloying (MA) of some pure elemental powders [1]. The formation of supersaturated, amorphous and metastble phases are common products of MA, however, it has been shown recently that the quasicrystalline phase can also be formed by MA process. [1,2]. The mechanism which is responsible for quasicrystalline formation is quite similar to that of amorphization by mechanical alloying [3,4]. During the process, the milled powder is deformed to a layered structure due to repeated deformation and cold welding. The true alloying occurs only, after the transformation of the layered structure to ultrafine matrix of powder at which the solid state interdiffusion reaction occurs. The ultrafine crystalline mixture of the elemental powder will have a high free energy [5]. Such free energy of the system can be reduced through the formation of the metastable, amorphous and quasicrystalline or forming crystalline intermetallic, mainly if the alloy system has a negative enthalpy of mixing [5].

In Al-18 at % Mn [6], as well as Al-Mn-Si (5 at % Si) [7] the quasicrystalline phase was observed after rapid solidification. However, it was not observed in binary Al-18 at % Mn when mechanical alloying of elemental powder was used·Moreover, the equilibrium intermetallic Al_6Mn was formed only during aging of the supersaturated Al-Mn solid solution produced by mechanical alloying [8].

In the present work, the $Al_{55}Mn_{20}Si_{25}$ atomic percent powder mixture is investigated after milling process in order to evaluate the effect of MA on the type of phase produced in this metal-metalloid system.

Experimental procedure

The mechanical alloying is performed in a high Cr steel ball mill using crystalline elemental powders with a particle size less than 100μm and purity of 99.9%. In each batch 10 gms of thoroughly mixed powder [55at% Al,20 at %Mn and 25at % Si] is used . The powder together with

steel balls were sealed in the water cooled ball mill using argon as the processing environment. The ball to powder weight is 10:1. The ball mill runs at a velocity of 360 revolution per minute for different time intervals up to 135 hours. The X-ray diffraction (XRD) studies are performed in a Siemens D-5000 diffractometer using CuK_{α} radiation. A Perken-Elmer7 (DTA) differential thermal analyser and differential scanning calorimetery (DSC) are used at a heating rate of 20K/min.

Results And Discussion

The x-ray diffraction pattern of as mixed powders of Al Mn Si are shown in fig (1), where all the major peaks of different elements are shown and used as reference for expected changes during milling process. In the early stage of milling, after 15 hrs, the diffraction pattern shows some changes by regard to the as- mixed state, fig (2-a), specially peaks intensities. The main reduction in intensities are observed for both Al & Mn element peaks. Also a strong new peak at d=0.3474 nm is noticed. This new line is found in all the milling times, up to 135 hrs. It is possible to index it to a type of the intermetallic phase $Mn_{12}Si_7Al_5$.

Further processing, 50 hrs, resulted in more peaks belonging to the intermetallic phase as well as an increase in their relative intensities as shown in fig (2-b). Moreover, it is possible to observe some of the apparently unreacted peaks; mainly (111) Al, (330) Mn and (111) Si, increase in their relative intensities with slight increase in d-value. Relative broadening of peaks are also observed for certain in the X-ray pattern, reflecting the effect of strain produced by the milling process. Some of the apparently unreacted peaks could be indexed, either, as a supersaturated solid solution of the Mn in Al, or as the intermetallic Al_6 Mn phase, with an orthorhombic unit cell in accordance with ASTM card number 6-665. Moreover, it is more likely that these peaks correspond to the supersaturated solid solution of Al, since the kinetics of MA process would favor usually the metastable supersaturated solid solution rather than the equilibrium Al_6 Mn. Accordingly, the aluminum solid solution in the mechanically alloyed conditions has a lattice parameter of 0.4018 nm. Only slightly smaller than the value of 0.4049 nm reported for pure Al. This could be due to the difference between atomic radii of both Al (0.1431 nm) and Mn (0.112 nm).

Therefore, the slight decrease in the Al solid solution parameter under the present conditions is justified.

The number of intermetallic peaks increases with further milling time and also the supersaturated solid solution lines are still persisting up to 90 hrs milling time. Figure (2-c) shows the diffraction pattern of the powder after 135 hrs MA and it is possible to observe that lines of both phases are almost disappeared in the background with exception of the main peak of the intermetallic phase. This could indicate that the supersaturated solid solution gives to a new amorphous phase, however, such transformation is not completed. The amorphous phase is provided by an increase in the background level and a halo region within the range of $20<2\theta<40$ on the diffractogram and the intermetallic peak over ridden the halo region.

The DSC results of MA powders heated at scanning tare 20K min^{-1} indicate the presence of two exothermic peaks at onset temperature of 538k and 817k, the first peak is less pronounced compared to the second one with $\Delta H_1 = 25.93$ J/g and $\Delta H_2 = 43.55$ J /g respectively . It is possible to attribute the first DSC peak to the decomposition of the supersaturated solid solution into the equilibrium intermetallic Al_6Mn phase such possibility is based on the transformation of the amorphous phase of Al-Mn-Si produced by rapid solidification on heating at 873 K [9].

One of the MA advantages is the heavy deformation which leads to structure refinement and the possibility of producing a nano-scale crystallites as can be seen in fig (3) which illustrates the fine size of MA powders up to 135 hrs. Figure (4) shows the variation of particle size with milling time for the main phase $Mn_{12}Si_7Al_5$. The unit cell size is determined, according to Scherres's formula [10]. The starting elemental powders have an average size less than 100 μm. The particle size is shown to vary between 20-14nm and decreases slowly during milling process. It is generally known that a nanocrystalline size material is characterized by both high strength and ductility Therefore, the bulk form of this ternary system of MA would be expected to show an improvement in both toughness and strength compared to the as cast state of the same system [11].

In general, the X-ray diffraction patterns recorded for the present MA powders show that, in the as milled conditions, only supersaturated solid solution and intermetallic phases with amorphization tendency at higher milling time. This result will emphasize the role of Si on the binary Al-Mn,

system. No intermetallic is formed in the binary system during mechanical alloying [8]. Moreover, it is reported that in the conventional as cast alloy of Al-Mn, silicon additions greatly accelerate the decomposition of the supersaturated solid solution of Al-Mn and enhance the precipitation of the ternary intermetallic phase [12]. Similar argument can also be adopted for present analysis in order to explain the reason behind the formation of the intermetallic phase under mechanical alloying conditions.

The transformation of the intermetallic phase with milling time into amorphous phase however, both phases are metastable, could be only understood in the direction of lowering the free energy of the transformation to quasicrystelline either in rapid solidification or during mechanical alloying [7] is not conformed by the present ternary alloy composition. The kinetic restrictions of MA process could be possible of preventing the formation of quasicrystalline phase in this metal–metalloid system.

Conclusions

The mechanical alloying behavior of the ternary AlMnSi have been examined in the present work and the following conclusions could be drawn out .

1- The formation of the metastable crystalline intermetallic phase $Mn_{12}Si_7Al_5$ with supersaturated solid solution are formed due to solid–state interdiffusion reaction.

2- A partial amorphous phase is prevailed after 135 hours.

3- The crystallite size of the $Mn_{12}Si_7Al_5$ is in the order of 20-14 nanometer.

4- Heating of MA powders has shown two exothermic peaks at 538 K and 817 K corresponding to the decomposition of the supersaturated solid solution and transformation of the partial amorphous phase, respectively.

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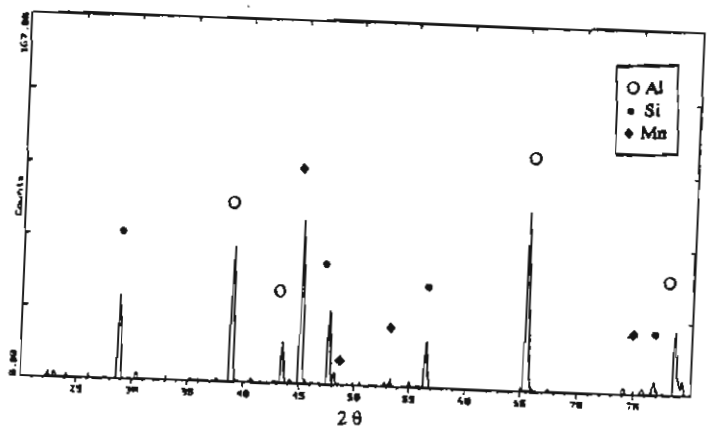

Fig (1) XRD pattern of elemental powders

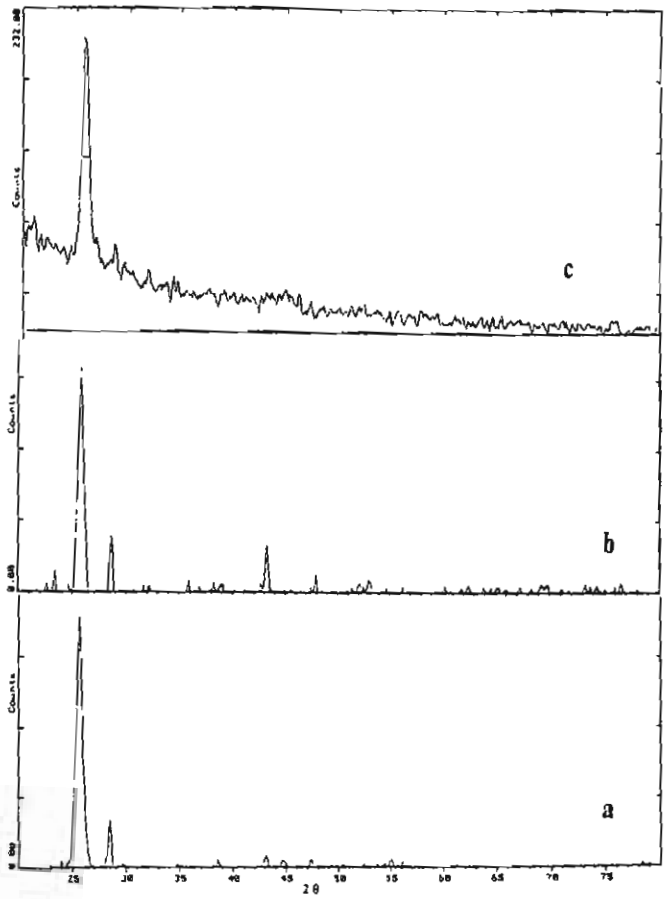

Fig (2) XRD pattern of milled powders.(a) 15 hrs, (b) 50 hrs and (c) 135 hrs.

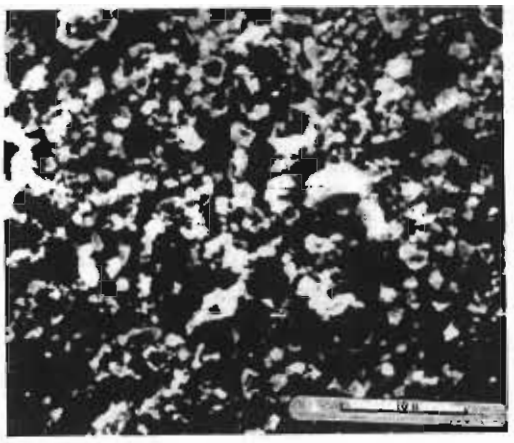

Fig (3) SEM micrograph for MA powder after 135 hrs.

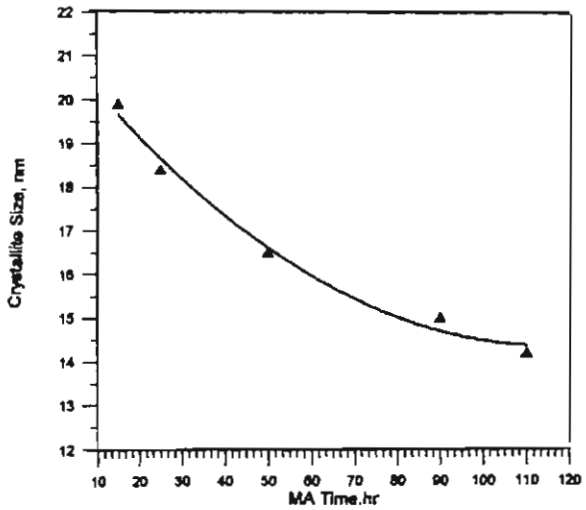

Fig (4) Crystallite size of mechanically alloyed $Al_{55}Mn_{20}Si_{25}$ at% Powders as function of MA time