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# MICROSTRUCTURAL DEVELOPMENT DUE TO MILLING OF RAPIDLY SOLIDIFIED Fe- 3Al- 5SI ALLOY

# التغير فى البنيه الميكروسكوبيه نتيجة لطحن سبائك الحديد-الألمنيوم-السيليكون المنتجة بالتبريد السريع

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الخلاصه:

تم دراسة التغير فى حجم وتركيب البنية الميكروسكوبية لسبائك الحديد-الألمنيوم-السيليكون المبردة تبريدا سريعا باستخدام الماسح الحرارى التفاضلى (DSC) والميكروسكوب الاليكترونى النفاذ والماسح وكذا الميكروسكوب الضوئى وذلك خلال مراحل الطحن المختلفة.

وقد أظهرت النتائج أن حجم كريات المسحوق النهائى بعد ٢١ ساعه من الطحن يقدر بحوالى ١ ميكرون بينما حجم حبيات البنيه الميكروسكوبيه فى هذه الكريات يقدر بحوالى ٥ نانوميتر بعد ٢١ ساعه من الطحن وقد اظهر الماسح التفاضلى الحرارى عدة تغيرات (طاردة للحرارة) ترجع الى الاسترجاع (Recovery) واعادة التبلور لمسحوق السبائك المشكلة بشدة.

## ABSTRACT

The modification and reduction of particle size of rapidly solidified shots of Fe- 3Al-5Si during ball milling is investigated. The reduction of the microstructure is monitored by differential scanning calorimetry and transmission electron microscopy. Optical and scanning electron microscopes have been used to investigate the metallographical and morphological changes of the ball-milled alloy powders at the several stages of milling. The results have shown that the end-product of Fe-3Al-5Si alloy milled for 21 hrs consists of uniform fine (1 μm in diameter) powder particles with spherical-like shape It is also shown that the ball milling technique reduces the particle size of the alloy powder to a nanometer scale (about 5 nm) after 21 h of milling. The differential scanning calorimetry has shown several exothermic peaks, which could be attributed to the recovery and the recrystallization of the severely deformed alloy powders.

## 1. INTRODUCTION

Ball milling (BM) may be referred to produce homogeneous composite particles with intimately dispersed, uniform internal structure. Since 1970 [1] the BM process has been used for preparing several dispersion-strengthened alloy powder [2-4]. Apart from producing the dispersed alloys, for their subsequent beneficiation, Koch et al. [5] employed that the BM technique for preparing an amorphous $Ni_{60}Nb_{40}$ alloy, starting from elemental powder of Ni and Nb, using a method so-called mechanical alloying (MA). This unique process leads to the formation of many advanced materials that can not be prepared by any other method, e.g. Al-Nb [6], Al-Ta [7], Ta-Cu [8] and Al-Hf [9] binary systems

Due to their unique atomic structure, nanometer-size materials have received great interest since being first reported by Birringer et al. [10]. These materials are usually prepared by the compaction of small metal clusters prepared by an inert gas condensation method. Such nanocrystalline metals and alloys are characterized by a high density of grain boundaries, that provides the possibility to study the properties of grain boundaries in more details than was accessible in the past.

In fact, Fe-Al-Si alloys have received attention because of their potential as low-cost, iron-based, high-temperature alloys, the oxidation resistance being provided by Al rather than the more critical alloying element Cr [11]. The Al content in Fe-Al binary alloys for satisfactory oxidation resistance has been found to be 8 wt. %. However, addition of Si to Fe-Al alloys enhances the oxidation resistance, a poor ductile Fe-Al-Si alloy is formed [11]. The ductility of bcc metals and alloys can be increased by reduction of the particle size because the slip distance of the dislocation is decreased and, thereby, the tendency for crack initiation is reduced. For such a purpose, the present study has been addressed in part to offer a powerful process for fabricating a nanocrystaline Fe-3Al-5Si alloy powders by BM technique. Although, with the advent of rapid- solidification-rate technology, the present work proves that it has now become possible to obtain, by BM, Fe-Al-Si alloys with greater homogeneity and extremely fine particle size.

## 2. EXPERIMENTAL PROCEDURES

The starting material of rapidly solidified Fe- 3Al- 5Si alloy shots were prepared at the West Beach facility of Pratt and Whitney, USA. Certain amount of these shots were charged in hardened tool-steel vial and sealed together with 440°C martensitic stainless-steel balls in a glove box under a purified argon atmosphere ( $O_2$ and $H_2O$ are less than 1 ppm ). The ball-to-powder weight ratio was controlled to be 10:1. The milling process was carried out at ambient temperature by mounting the vial on a high energy ball mill of type Spex-Mixer/Mill, model 8000. The milling process was interrupted at selected intervals ( starting with 1 hr ) and a small amount of the milled powders was taken out from the vial in the glove box. The milled powders were characterized by differential scanning calorimtry (DSC) Dupot model 9900 under argon flow with heating rate of 20°C/min. The morphology of the powders were studied by optical microscopy, scanning electron microscopy (SEM) using 30 KV microscope and transmission electron microscopy (TEM) using 200 KV microscope.

## 3. RESULTS AND DISCUSSION

### 3.1. Morphological and Metallographic Changes with the BM Time:

SEM technique was used to follow the changes in the size and the shape of Fe- 3Al- 5Si powder particles during the several stages of milling. Figure 1 shows the SEM micrographs of the milled powder particles at the early [Fig.1(a)] and after the final [Fig.1(b) ] stages of milling. Obviously, after 2 hrs of BM time, the powder particles are bulky and have irregular shape with average particle size of 15 μm, as illustrated in Fig. 1 (a). The microstructure examination of a polished and etched surface of the alloy powder indicated that the particles have grain-like morphology, as presented in Fig. 2(a). During the progress of the BM process, the assemblage particles are subjected to several forces such as impact and shear forces that generated from the milling media so that the powder particles are continuously disintegrated during the BM time. At the end of the BM process ( 21 hrs ) the powder were homogeneous in shape with spherical-like morphology and uniform in size (about 5 μm in diameter), as presented in Fig. 1(b). Moreover, the grain-like morphology of the powder particles has already disappeared and the individual particles have fine structure as shown in Fig. 2(b).

3.2. Structural Changes with the BM Time:

The local structure of Fe-3Al-5Si alloy powder has been investigated by TEM technique. Figure 3 represents the (a) bright-field image (BFI) and (b) dark-field image (DFI) together with the corresponding selected area diffraction patterns of (SADPs) for ball milled Fe-3Al-5Si alloy powders milled for 2 hrs and 21 hrs, respectively. At the early stage of ball milling (2 hrs) the powder contains large polycrystalline particles with sharp boundaries, as displayed in Fig.3(b). Since the grains are quite coarse, the SADP shows sharp spot rings, as illustrated in Fig.3(b). Increasing the milling time leads to decrease the crystalline size of Fe-3Al-5Si alloy and the powder particles have fine cell-like morphology with average particles size of about 5 nm in diameter, as displayed in Fig. 3(C). The corresponding SADPs [Fig.3(d)] reveal that the orientation of neighboring particles is random.

The TEM technique allows us to determine the crystalline size of the Fe-3Al-5Si alloy powders during the ball milling process. Figure 4 displays the relationship between the particle size of the alloy powder and the milling time. Remarkably shows, the particle size of Fe-3Al-5Si alloy monotonically decreases with increasing the milling time to great value of about 5 nm in diameter after 16 hrs. This value dose not change with longer milling time, as long as 21 hrs, as represented in Fig. 4. It is worth noting that the distribution of the particle sizes becomes narrow with increasing the milling time, indicating that extended ball milling possesses a uniform size distribution for longer milling times.

3.3. Thermal Stability

In addition to the morphological and structural investigations, the thermal stability of ball milled Fe-3Al-5Si alloy powders have been examined by DSC. Figure 5 shows the typical DSC traces for the ball milled alloy powders as a function of the milling time. A single broad exothermic peak appears at about 418°C in the SDC curve for the as-received (no milling ) alloy powders, as illustrated in Fig. 5(a). This peak is attributed to the releasing of the strain energy in the powder particles. Increasing the milling time leads to the increase of the area under this exothermic reaction, suggesting an increase in the strain energy that took place during the ball milling process of Fe-3Al-5Si alloy powders After 2 hrs of the milling time, a broad exothermic reaction occurred at low temperature (about 109°C) that could be due to deformed powder the crystal growth, as presented in Fig. 5 (d). Contrary to this, a high temperature exothermic reaction starts to appear at about 650°C for the sample milled for 5 hrs, as displayed. This high temperature peak becomes more pronounced and sharp at the final stage of milling (21 hrs), as shown in Fig. 5(b). We attribute this exothermic peak to recovery and recrystallyzation processes and/or nano-phase coarsening.

## 5. CONCLUSION

The modification and reduction of particle size in rapidly solidified shots of Fe-3Al-5Si alloy during ball milling has been reported. The microstructural change were monitored by differential scanning calorimetry and transmission electron microscopy. Optical and scanning electron microscopes heve been used to understand the metallographical and morphological changes of the ball-milled alloy powders at the stages of milling. The results have shown that the end-product of Fe-3Al-5Si alloy milled for 21 h consists of uniform fine (1 μm in diameter) powder particles with spherical-like morphology. It is also shown that the ball milling technique reduces the grain size of the alloy powders to nanometer scale (about 5 nm) after 21 h of milling.

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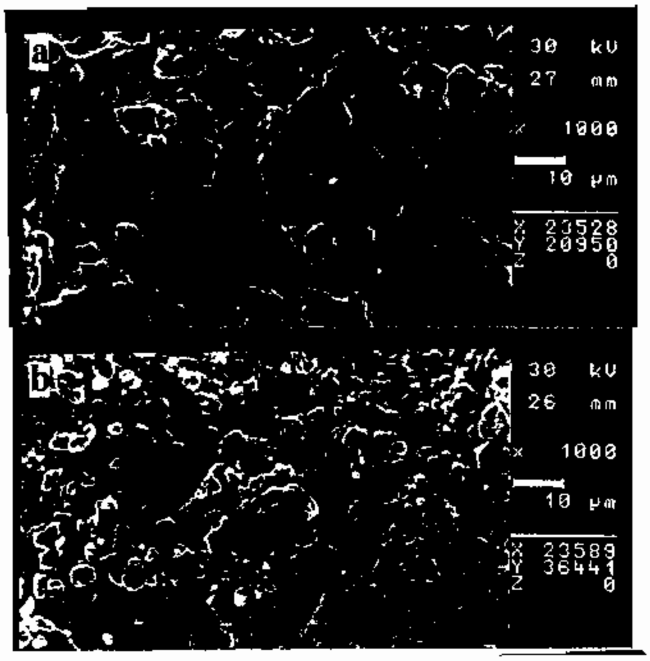



**Fig. 1.** SEM micrographs of Fe-3Al-5Si alloy powders milled for (a) 2 h, and (b) 21 h of the ball milling times.

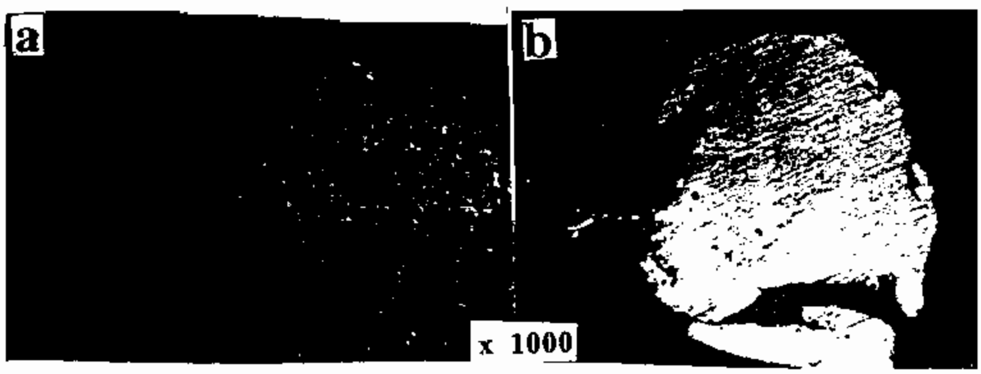



**Fig. 2.** Optical micrographs for the polished surface of Fe-3Al-5Si alloy powders milled for (a) 2 h and (b) 21 h of the ball milling times.


a
b

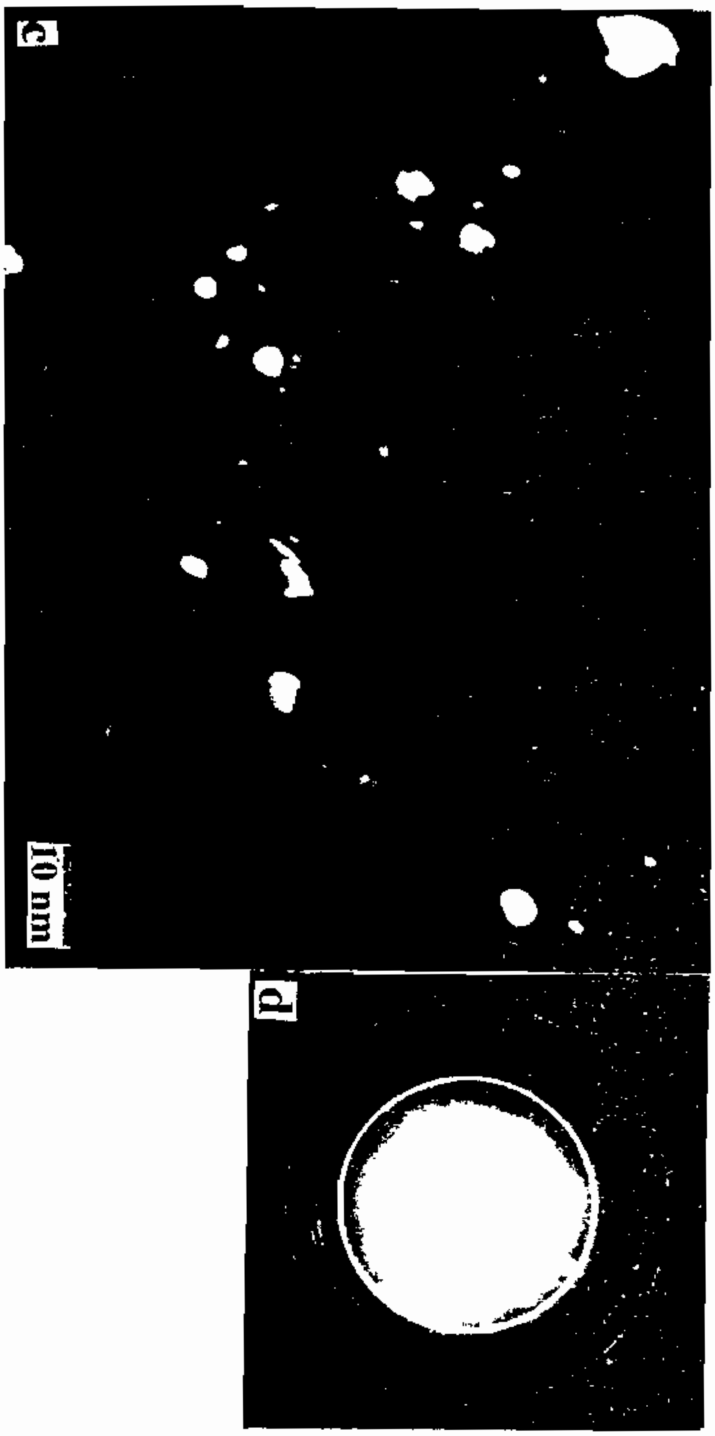



**Fig. 3.** Detailed TEM observations for Fe-3Al-5Si alloy powders after selected (a and b) 2 h and (c and d) 21 h of the ball milling times. The BFI and/or DFI are shown together with SADP.

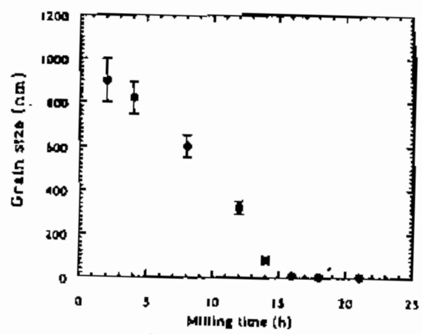
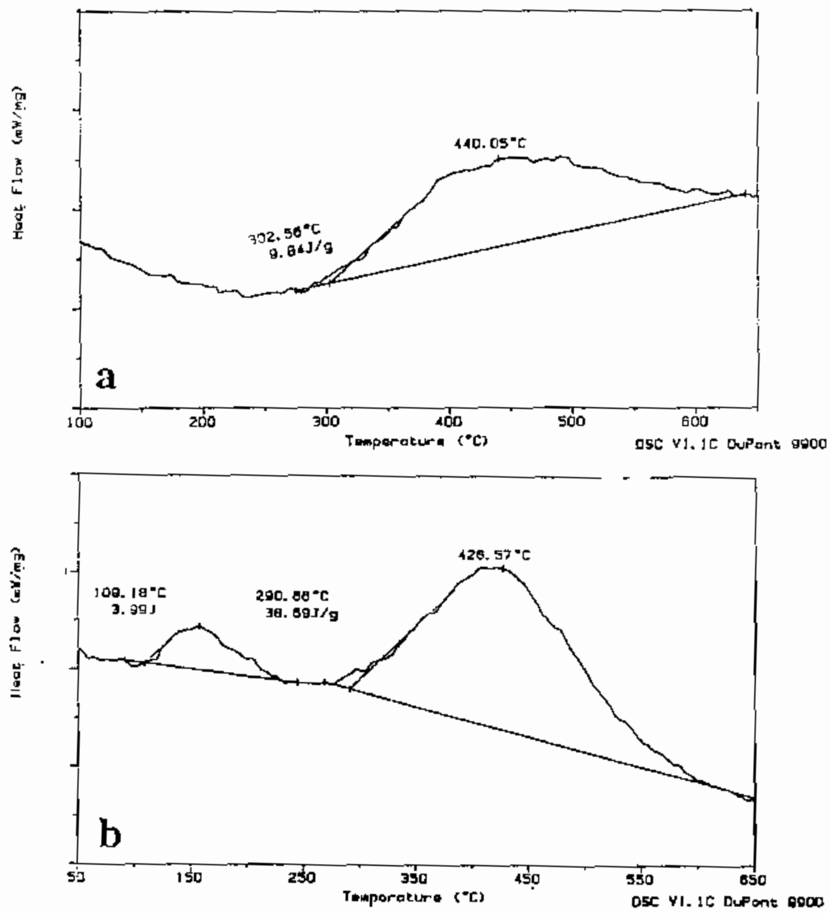
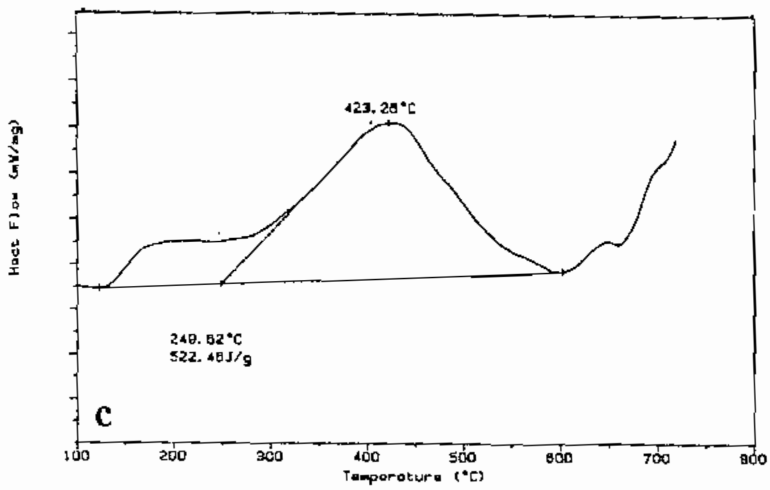



Fig. 4. Grain size distribution of Fe-3Al-5Si alloy powders as a function of the ball milling times.





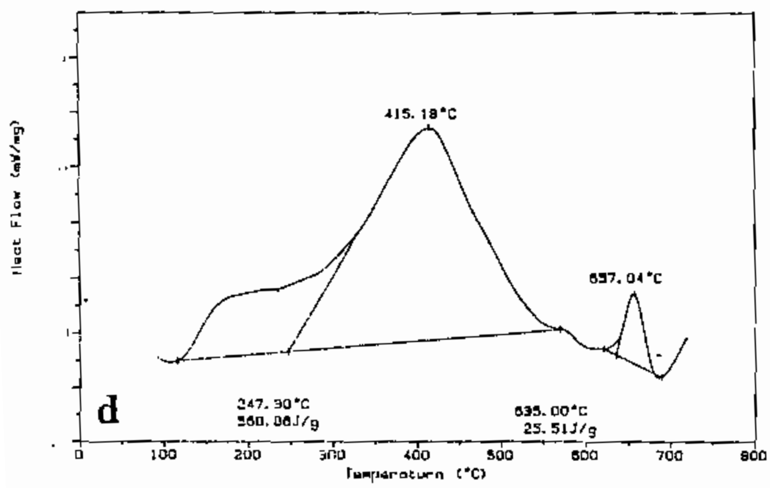




Fig. 5. DSC traces of Fe-3Al-5Si alloy powders after (a) 0 h, (b) 2 h, (c) 5 h and (d) 21 of the ball milling times.