

Effect of Cr to Fe on the Solid Solubility, Lattice Parameter and Strain of Fe₈₀Cr₂₀ Alloy Powder

Dafit Feriyanto^{1,a}, M. I. Idris^{1,b}, Darwin Sebayang^{2,c}

¹ Faculty of Mechanical and Manufacturing Engineering, University Tun Hussein Onn Malaysia (UTHM), Parit Raja, BatuPahat, 86400 Johor, Malaysia

² Faculty of Mechanical Engineering, MercuBuana University, Indonesia

^adafitferiyanto@yahoo.co.id, ^bizwana@uthm.edu.my, ^cd_sebayang@hotmail.com

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Abstract. α -Fe-Cr phase was investigated using formula Fe₈₀Cr₂₀. Ball milling process and ultrasonic technique is successfully done to develop solid solubility and improve homogenous, respectively. However, the effect of the Cr to Fe powder is not completely investigated using combination of its process. Ball milling is conducted by milling time of 60 hours and ultrasonic technique were carried out at ultrasonic time of 3 h, 3.5 h, 4 h, 4.5 h and 5 h. From the strain effect analysis is obtained that the strain increased with crystallite size decreased and broad peaks due to the micro strain that is obtained from the increasing d-spacing. The solid solubility and lattice parameter of the material relatively increased from the untreated sample to treated samples with the highest solid solubility of 62.1% and highest lattice parameter of 3.091 nm which is located at the milled and UB 4.5 hours. It is caused the temperature increased that effect to the higher diffusion of the atom. Therefore, the combination treatment is highest promote to improve the properties of the metallic materials.

1. Introduction

Interaction of Cr powder in Fe powder has become an important knowledge field in engineering steels for making steel and controlling the properties [1]. On the Fe-Cr phase diagram, α -iron phase plays a critical property in ferritic steel product. However, slightly data which describes those fields specifically. Many challenges of α -iron chromium phase on the experimental action such as low solubility Cr. In order to achieve the true equilibrium, long time treatment should be conducted [1]. α -Fe-Cr phase has high stability in high temperature operation and improves some useful properties. That phenomenon has become a highly desirable effect. Different with σ -Fe-Cr phase, high corrosion resistance in high temperature has been observed while it is thermodynamically unstable. This phenomenon has unwanted properties [2]. Therefore, it is very interesting to develop σ -Fe-Cr phase for investigation of physical properties and need as a reference system. This research investigates the FeCr alloy for interconnect application. According to the Fe-Cr diagram Fig.1, one of the formulas for developing α -Fe-Cr phase was achieved using different compositions of Fe and Cr. According to [3, 4] the formula is Fe₈₀Cr₂₀ alloy in order to create the α -Fe-Cr phase and the desired properties.

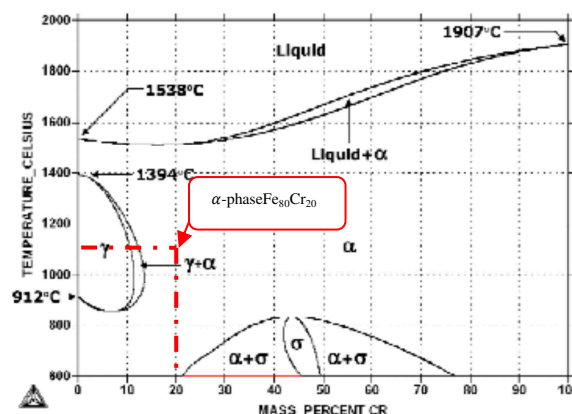


Fig.1 Phase diagram of Fe-Cr system [5]

Lattice spacing usually shows a few thousand atoms distance. Lattice parameter is based on quantity of most solid solution objects. Analysis of lattice parameters as a basis for a solution has become a topic in solid state physics and materials science. The analysis uses Vagard's formula as considered [6 - 8].

The differences of the solubility product are depending on the solid phase. Large monocrystal have the constant thermodynamic solubility and it will be increased by the monocrystal size decreased because the surface energy also increased. The solid solubility relative increased with the temperature increased is named as endothermic reaction. Meanwhile, the exothermic reactions occur when the solid solubility decreased with temperature increased. These phenomenon is due to the temperature effect on the by recrystallization process, change of the enthalpy and entropy [9-12]. According to [13-15] that high solid solubility has been achieved in small crystallite size, high strain material and high lattice parameter. Crystallite size has increased by decreasing the strain. This phenomenon is related to the strength and fatigue. Strain of material has increased and indicated that the strength and fatigue properties have increased. The solid solubility increased shown by the increasing of kinetic energy which is allows the solvent molecules are more effective to break apart the solute molecules that are held together by intermolecular attractions. From the researcher above is obtained from the ball milling process. Introducing the ultrasonic technique is successfully done by many researchers [16-20] to improve the homogenous. However, the combination of it is not complete investigated.

2. Methodology

The treatment was conducted by using ultrasonic technique, mechanical alloying using high energy ball milling and combination treatment (ball milling combined with ultrasonic technique). The sample designation is listed in Table 1.

Table 1 Various sample designation

| Sample Designation | Treatment detail |
|---------------------|---|
| Raw material | Raw material |
| UT 3 h | Fe ₈₀ Cr ₂₀ with Ultrasonic time of 3 hours |
| UT 3.5 h | Fe ₈₀ Cr ₂₀ with Ultrasonic time of 3.5 hours |
| UT 4 h | Fe ₈₀ Cr ₂₀ with Ultrasonic time of 4 hours |
| UT 4.5 h | Fe ₈₀ Cr ₂₀ with Ultrasonic time of 4.5 hours |
| UT 5 h | Fe ₈₀ Cr ₂₀ with Ultrasonic time of 5 hours |
| Milled 60 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours |
| Milled and UT 3 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours and ultrasonic time of 3 hours |
| Milled and UT 3.5 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours and ultrasonic time of 3.5 hours |
| Milled and UT 4 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours and ultrasonic time of 4 hours |
| Milled and UT 4.5 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours and ultrasonic time of 4.5 hours |
| Milled and UT 5 h | Fe ₈₀ Cr ₂₀ with ball milling time of 60 hours and ultrasonic time of 5 hours |

Refer to Table 1 that the ultrasonic technique was carried out using various times of 3 to 5 hours with different time of 0.5 hours. The ball milling process was carried out using fixed milling time of 60 h. Meanwhile, the combination treatment is combination between milling time of 60 h and various ultrasonic times. Raw material is formulated as Fe₈₀Cr₂₀ alloy powders.

2.1 Measuring lattice parameter

The distance equals an integer multiple of the wavelength is called diffraction order. Bragg's law is used to obtain the lattice spacing of a particular cubic system through the relation as follow:

$$a = d \cdot \sqrt{h^2 + k^2 + l^2} \quad (1)$$

where, h, k, l = The miller indices of the diffracting plane
 a = The lattice parameter of the cubic material
 d = Interplanar spacing

Cu-K α ray were produced where the step size of 0.017 (2θ) was used for a range of 10-90 $^\circ$ (2θ) for lanthanum hexaboride (LaB $_6$). Traditionally, X-Ray diffraction has been used for describing the crystallite size change and disorder/defect in the crystal structure [22]. The mechanism of the X-Ray Diffraction is to analyze the samples as shown in Fig.2.

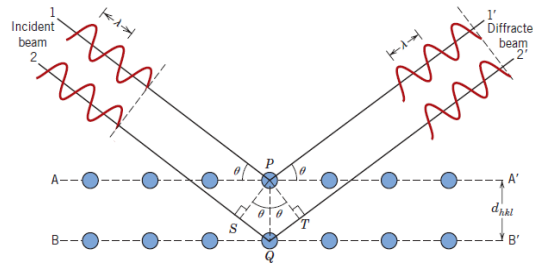


Fig.2 X-Ray Diffraction [21]

Three dimensional space are described with reciprocal axes x, y and z to eliminate the effects of texturing. The diffraction intensity is a function of the scattering angle 2θ or scattering of vector q which make it independent. Study lattice parameter has been studied by some researcher such as [23, 24]. They found that the highest and lowest shear stresses having the largest and smallest lattice parameter, respectively.

2.2 Measuring solid solubility

Solid solubility of the sample designation of Fe $_{80}$ Cr $_{20}$ alloys powders could be estimated by the Vegard's formula as showed in equation number 2 [25] and this equation or principle has been used for many researcher [6, 23, 26]. Solid solubility is introduced to determine the ability of one substance and to combine with another as well as to form a homogenous. Besides, solid solubility is needed to make the solution with another substance [25]. Phase stability of the Fe-Cr ally can be predicted from the deviation of the lattice parameter from Vegard's formula as reported by [26] on the atomic level.

$$x_{ss} = \frac{(a_{ss} - a_1)}{(a_2 - a_1)} \quad (2)$$

where, a_{ss} = lattice parameter of solid solubility
 a_1 = lattice parameter of pure solvent element
 a_2 = lattice parameter of solute element
 x_{ss} = solid solubility

3. Result and discussion

Combination process (milled and UB) were most effective to develop nanocrystalline Fe $_{80}$ Cr $_{20}$ by compared the milled 60 h samples. It due to the high energy ball milling which was blended the powder up to 60 h lead to the powder size which were significant to reduce the powder size and the vibration from the ultrasonic treatment lead to the homogenous powder size has improved. The influence of the strain on the diffraction peaks and the crystallite size is shown in Fig.3.

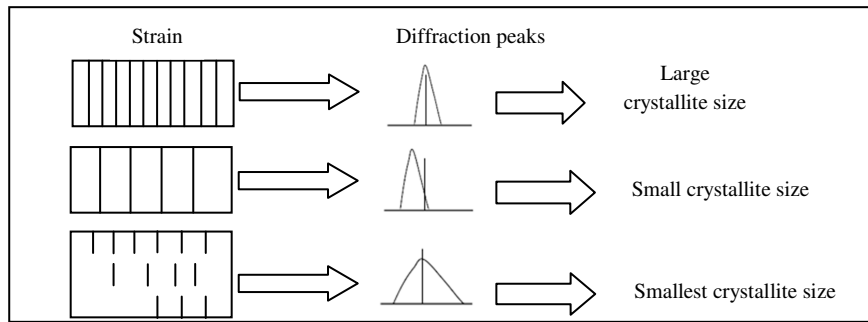


Fig.3 Effect of strain on the diffraction peaks

The higher strain of material lead to the broader diffraction peaks. It is because the d-spacing of the materials in increase which is caused by micro-strain. Sharper diffraction peaks are shown in the larger crystallite size. It is possible caused the larger crystallite size led to smaller lattice parameter. The solid solubility (x_{ss}) vs. Lattice parameter (a) of the sample designation is shown in Fig.4.

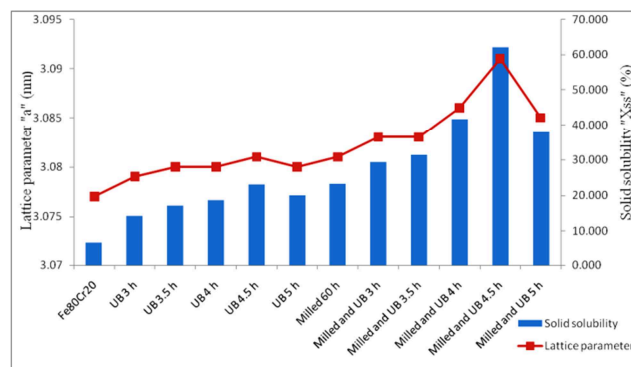


Fig.4 Lattice parameter vs solid solubility of the treated and untreated samples

Fig.4 shows the pattern of lattice parameter and solid solubility is one line. It is due to the determining solid solubility which depends on lattice parameter. Therefore, if the lattice parameter is increased, the solid solubility is increased. Solid solubility of the combination (milled and UB) samples are better than raw material, UB samples and milled 60 h sample. It was proved by solid solubility of the combination treatment (milled and UB) increase up to 89% when compared with raw material, 77.3 % when compared with ultrasonic (UB) sample and 63% when compared with milled 60 h sample. It shows the combination treatment was effective to produce the solubility of Cr powder to Fe powder. Developing solid solubility is dependent on the temperature. According to [27] that the Cr powder has higher thermal resistance and it used as coating material. Therefore, the significant effect of the Cr powder in the $Fe_{80}Cr_{20}$ alloy is shown when ball milling process. When the $Fe_{80}Cr_{20}$ dissolves in a ball milling machine and ultrasonic machine temperature has increased, the average kinetic energy of the $Fe_{80}Cr_{20}$ alloy powder also increases. The increasing of kinetic energy allows the solvent molecules are more effective to break apart the solute molecules that are held together by intermolecular attractions. The solid solubility was investigated by [14, 15, 27] that the ball milling effective to develop solid solubility of Cr to Fe powder because the high energy kinetics and the balls slugging the sample powder during ball milling process. The solid solubility when ball milling process is increased because the diffusion of the component atom increased. It is caused by larger volume friction of the atom samples in the grain boundaries [27, 28]. The vibration in ultrasonic process and the ball collision on sample in ball milling process has increased and this lead to less able to hold together.

4. Conclusion

Higher solid solubility, lattice parameter and strain is successfully obtained using combination treatment between ball milling and ultrasonic technique. Moreover, effect of the Cr to Fe is closely

improve the corrosion resistance promoted by α -Fe-Cr phase. It approved when the ball milling process, the temperature is raise lead to the larger volume friction of the atom of α -Fe-Cr.

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