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Theme of the Paper: Microalloying in ODS Steel

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EFFECT OF LOW AMPLITUDE IRRADIATED ULTRASONIC METHOD OF CHARACTERIZATION MICROALLOYING Fe-Cr-Y_2O_3

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Abstract: *ODS Steel (Oxide Dispersion Strengthened) is an alloy with good corrosion and high temperature resistance. The utilization of ultrasonic irradiation method in toluene solution is a new method giving benefits like producing fine grain-size, forming micro-alloying on powder, and minimizing the oxide phase formation. This research investigates the influence of low amplitude on the reduction of the particle size and the formation of Fe-Cr microalloying on Fe-20Cr-1*Y_2O_3 *powder preparation. The powder was divided into 3 samples with ultrasonic irradiation and various amplitude of 20%, 30% and 40%. After that, each sample was characterized by SEM, EDS and XRD. This research shows that increasing the amplitude will decrease the particle size with the value of sample 20% > sample 30% > sample 40% is 4.60 µm>3.71 µm> 3.60 µm. The highest composition value of Fe-Cr is reached on the amplitude of 20% to the composition value of sample 20%: sample 30%: sample 40% is 81.9%: 81.2%: 64.7% without the oxide formation. The size of crystallite Fe-Cr sample 20%: sample 30% : sample 40% size of 256.3 nm: 198.0 nm: 184.3 nm.*

Keywords: *ODS, ultrasonic irradiation, amplitude, microalloying, cavitation*

1. Introduction

ODS (Oxide Disperse Strengthened)steel is generally synthesized by mechanical alloying[1] method. In 1950, the making of Fe-Cr combination started from secondary steel making in furnace. The study continued in 1950-1960, using vacuum arc degassing by blowing Ar gas, so that there is mixing in thefurnace ladle [1,2]. With increasingly modern technology, ODS steel synthesis then used planetary or attritor ball mill for powder mechanical alloying and then using High Energy Milling, Hot Isostatic Pressing[1-4], hot extruded at 1150 ºC, attritor ball mill by blowingargon gas, High planetary ball mill under argon atmosphere[4]. Mechanical alloying treatment often creates oxide and the resulting alloy isn't homogenous enough.

For an alternative, ultrasonic treatment is a potential technique in making more homogenous powder and preventing oxide formation. Ultrasonic irradiation method is a relatively new method and is an effective alloy forming technology [5-7]. Cavitation energy is an energy produced by ultrasonic wave and used to form Fe-Cr-Y_2O_3 microalloying [6-7]. ODS steel is developed by evenly spreading fine oxide particles, and nanometer-sized Y_2O_3oxide particles. Material with oxide particle dispersion has advantages because the oxide particles are spread evenly across matrix. Homogenous and evenly spread oxide particles will improve the endurance of creep[8-10].

2. Methodology

The allow powder materials were Fe with particle size around 3 µm, Cr with particle size around 23 µm, and Y_2O_3 with particle size around<50 nm. The powders are Aldrich products with 99.9% purity. Toluene with 90% purity was also used as powder media in ultrasonic irradiation treatment. 6 gram of *microalloying* (Fe-Cr-Y_2O_3) material sample was weighed. The composition of Fe powder was 79%, Cr powder 20%, and Y_2O_3powder 1%. Then, the powder sample was inserted in glass beaker and toluene solution was added. The sample was given ultrasonic irradiation treatment for 30 hours (Ultrasonic Horn type Autotune series "High intensity Ultrasonic Processor", sonic vibracell amplitude, model VCX 750, TI horn with 20 KHz frequency) with 20%, 30%, and 40% amplitude variations to determine optimum amplitude in Fe-Cr-Y_2O_3 microalloying fabrication.

Morphological test and analysis of the microstructure composition of Fe-Cr used Scanning Electron Microscopy (SEM) equipped with Energy Dispersive Spectroscopy (EDS) JEOL, JESM 6510LA using 20 keV energy. Composition test was made at 3000x magnification and continued by more specific particle observation at 10.000x magnification.

Identification of phases Fe, Cr, and formation of new phase Fe-Cr were analyzed by Simadzhu XD-610X-Ray Diffraction (XRD). XRD measurement was perford by Cu Kα radiation (λ=0.15406 nm). To analyze the resulting Fe, Cr, and Fe-Cr cubic structures,cubic unit cells was calculated by Bragg's law which is:

$$\left(\frac{\lambda}{2a}\right)^2 = \frac{\sin\theta}{h^2 + k^2 + l^2}$$

Where λ is Cu Kα wavelength, *a* is the distance between atoms from the constituent cell unit, *h*, *k*, and*l*are miller indices of diffraction fields, and θ is the angle between the beamed radiation rayand diffraction field.

$$d = \frac{n\lambda}{2\sin\theta}$$

Where λ is Cu Kα wavelength, *n* is integer, *d* is the crystal lattice distance between atoms, and θ is the angle between the beamed radiation ray and diffraction field.

To analyze lattice parameter, percentage of mass of powder constituent phase, and crystal structure of XRD test, Rietveld and Warren-Averbach method [11,12] was used in MAUD software [13]. Curve smoothing method was used to adjust the parameter of the structure produced by XRD test with crystal structure model which had crystal symmetry form of each element. Ideal curve smoothing is indicated by goodness of fi tvalue which is defined to be ideal when it's $x \leq 1.00$ [11]. Goodness of fit calculation is defined as:

$$S = \frac{R_{wp}}{R_{exp}}$$

Where S is goodness of fit value ($x \leq 1.\ 00$), R_{wp} is weighted profile reliability-factor value, and R_e expected reliability-factor value.

3. Result and Discussion

3.1 The effect of amplitude on Fe-Cr particle formation

Figure 1 is the result of SEM test of alloy material powders. Figure 1 (a) is Fe powder with form resembling ball and isaround 3 µm. Figure 1 (b) is Cr with irregular form and is around 20 µm. Meanwhile, Figure 1 (c) is Y_2O_3 oxide particle with nanoparticle size (<50 nm).

Based on figure 2, figure3, and figure 4, the particle morphology resulting from ultrasonic irradiation have changed from the initial particle morphology. The phenomenon indicated that ultrasonic treatment can change the morphology of powder particle. The ultrasonic wave transmitted in toluene media can form tension and stretch [14] on powder. These density and stretch can cause the particles to collide because the particles move to all directions [15] and also can cause cavitation nucleation [16,17]. This cavitation nucleation is also caused by vapor and gas inside toluene media, if the pressure of vapor and gas are equal with the pressure around it then cavitation will form. Increased amplitude can increase the intensity and pressure working on particle [18]. Increased time and pressure cause cavitation collapse [19] with core explosion followed by *microjet* formation at 100 m/s and 1000 atm pressure [16,20]. The explosion is *in situ* and is localized in very small point. *Microjet* colliding with particle surface will increase surface reactivity [16,20]. If there is perfectly elastic collision, there will be friction between particles and causing reduction of grain size. If there is imperfect elastic collision [21], there will be unification of particles called powder agglomeration.

In figure 2, figure3, and figure 4, it's detected that the percentage of Fe and Cr compositions aren't very different in several different EDS test positions. It's believed that ultrasonic irradiation treatment caused the formation of Fe-Cr particle. Moreover in figure 2, Y_2O_3 oxide particle is detected, proving that ultrasonic irradiation can disperse particles well.It's because ultrasonic wave or cavitation collapse causes particle to be dispersed to all [22].

Table 1 The composition of the ultrasonic irradiation powder treatment element with an amplitude of 20%, 30%, and 40%

Perlakuan sampel	Posisi	Fe (wt%)	Cr (wt%)	Y_2O_3 (wt%)
20%	1	48.73	45.795	5.475
	2	83.51	16.49	0
	3	51.26	45.59	3.15
30%	1	39.52	60.48	0
	2	53.804	46.196	0
	3	46.548	53.542	0
40%	1	33.296	66.704	0
	3	58.443	41.557	0
	2	54.005	45.995	0

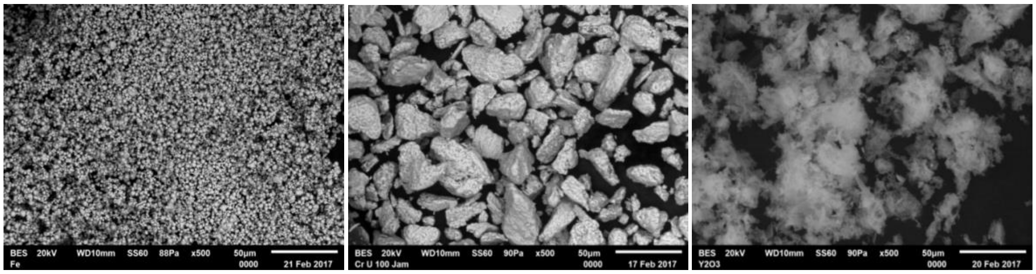

Figure 1 (a) Fe powder precursors, (b) Cr powder precursors, and (c) precursors Y_2O_3

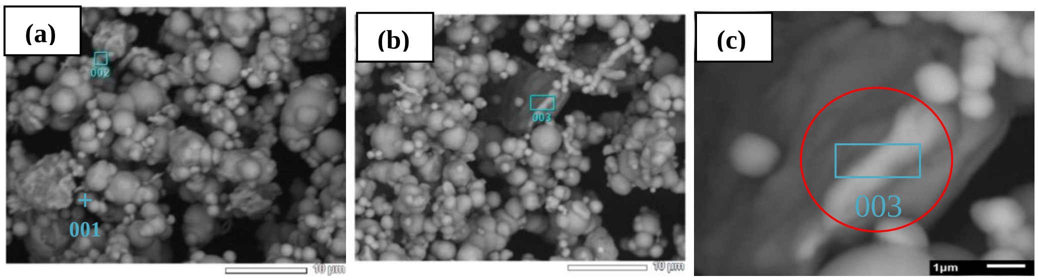

Figure 2 SEM sample of 20% amplitude ultrasonic irradiation, (a) position 001 and position 002 with magnification 3000x, (b) position 003 with magnification 3000x (c) magnification 10.000x position 003

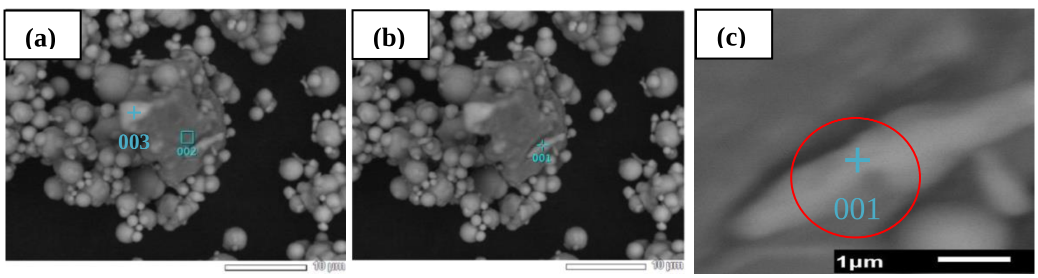

Figure 3 SEM ultrasonic irradiation sampling amplitude 30%, (a) position 002 and position 003, (b) position 001 with magnification 3000x, and (c) magnification 10.000x position 001

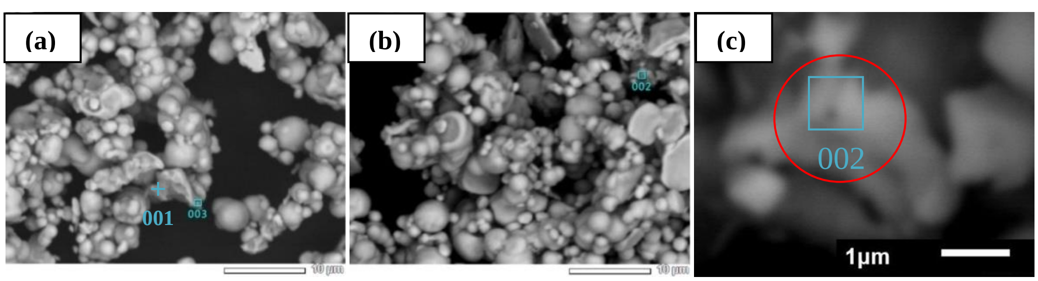

Figure 4 SEM sample of 40% amplitude ultrasonic irradiation, (a) position 001 and position 003 with magnification 3000x, (b) position 002 with magnification 3000x, (c) magnification 10.000x position 002

Ultrasonic irradiations with 20% and 30% amplitude variations produce complete Fe-Cr *microalloying*. On the other hand, 40% amplitude ultrasonic irradiation produces partial Fe-Cr *microalloying*. It's predicted that large amplitude causes larger pressure and more energy of the collision on particle tends to be transmitted for particle reduction than with lower amplitude.

3.2 The effect of amplitude on particle size

From the picture of the result of SEM test, average particle size was calculated by software ImageJ for samples with 20%, 30%, and 40% amplitude treatments, producing comparison of particle sizes as in figure 5.

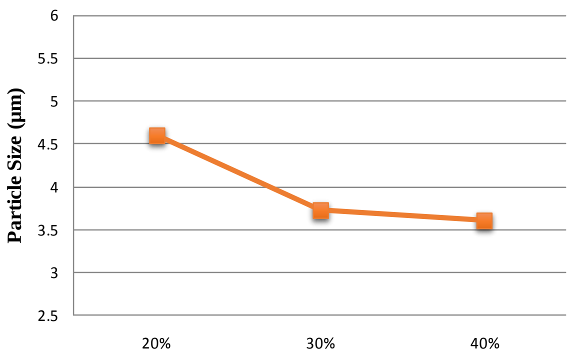

Figure 5 Comparison of particle size of ultrasonic treatment 20%, 30%, and 40%

Ultrasonic treatment will cause collision between particles due to energy from density and stretch in toluene media [14] and cavitation collapse, so there is reduction of particle size. Increased amplitude of ultrasonic irradiation will create chance of cavitation collapse and bigger *microjet* [20,23]. They cause larger reduction of particle size. The figure shows decrease of particle size along with increase of amplitude.

3.3 The effect of amplitude on Fe-Cr phase composition

Figure 6 is a graph of the result of curve smoothing of XRD test on all samples by software MAUD. From the result of curve smoothing of XRD test, lattice parameters values are obtained, showing the formation of BCC structures in all samples. It's confirmed bycloseness between lattice parameters from curve smoothing and JCPDF data (Fe=PDF#00-0060696, Cr=PDF#00-001-1250, Fe-Cr=PDF#1523982). The lattice parameter value of Fe-Cr is between the values of Fe and Cr, showing that new phase is formed as stated by Vegard's Law [24].

Table 3 shows phase composition formed in every experiment sample. In ultrasonic irradiation treatment sample (20%, 30%, and 40% amplitudes), there are three Fe, Cr, and Fe-Cr phases. The sample without ultrasonic treatment only forms Fe and Cr phases. The most optimal Fe-Cr microalloying percentage is 20% amplitude for 30 hours. Fe-Cr phase composition will decline along with increased amplitude.

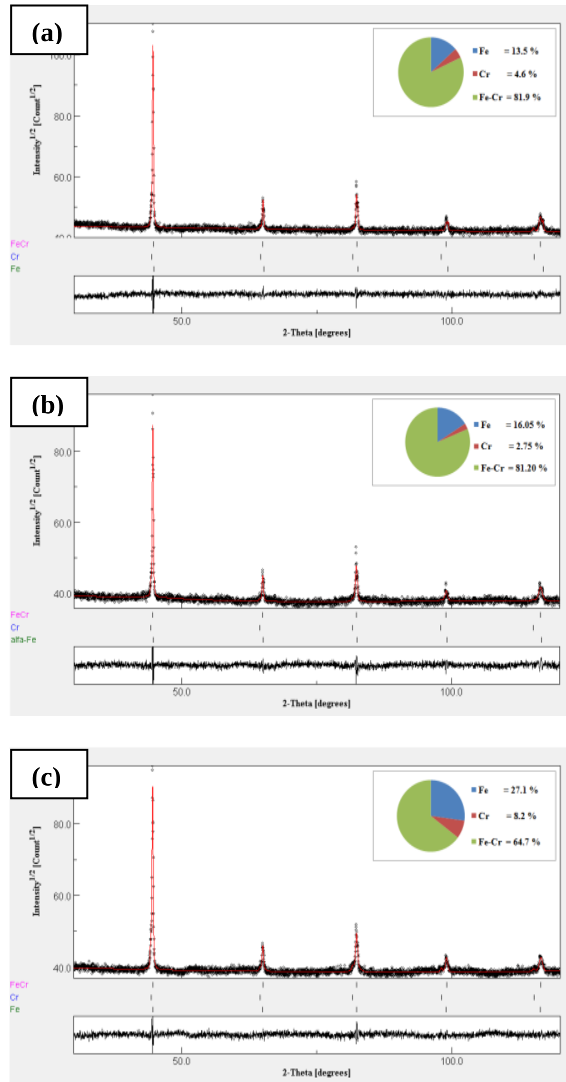

Figure 6 (a) the graph of the XRD curve of the sample 20%, (b) the sample 30%, and (c) the sample 40%

3.4 The effect of amplitude on crystallite size

The formation of *microjet* from cavitation collapse will cause crystallite sizereduction [7,18]and some crystal defectssuch as vacancy, dislocation, andgrain boundaries[25]. Figure 7 shows that the bigger the ultrasonic irradiation amplitude, the smaller the crystallite size. It's because increased amplitude also causes environmental pressure to be bigger, so chance of cavitation collapse is bigger. The calculation result shows declining trend of the values of 20%:30%:40 amplitudes which are256.38 nm: 198.02 nm: 184.34 nm, respectively.

Table 2 Summary of lattice parameters and phase percentages of samples 20%, 30%, and 40% from the results of rietvident refinement

Phase	Lattice parameter a=b=c(Å)				Mass fraction (%)		
	References	20%	30%	40%	20%	30%	40%
Fe	2.8664	2.8593	2.8607	2.8665	13.5	16.05	27.1
Cr	2.895	2.8861	2.8837	2.8858	4.6	2.75	8.2
FeCr	2.868	2.8668	2.8628	2.8651	81.9	81.2	64.7

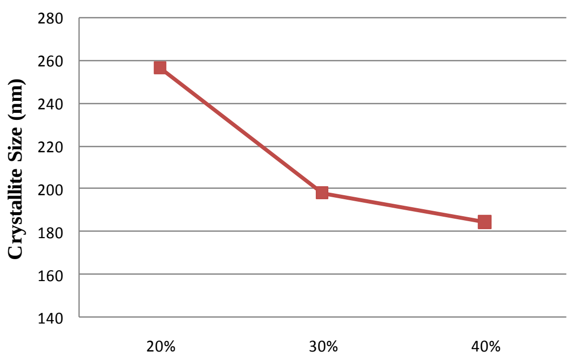

Figure 7 Comparison of Crystallite size Fe-Cr

4. Conclusion

This research has successfully made Fe-Cr microalloying using ultrasonic irradiation method. Cavitation bubble growth will cause cavitation explosion, followed by microjet formation. The microjet is used to reduce particle size or merge several particles. Particle merging is caused by collision between particles. During the process, the reactivity of Fe and Cr surfaces will increase, then Fe-Cr microalloying may be formed. 20% amplitude is the optimum amplitude to form Fe-Cr microalloying because it produces81.9%Fe-Cr. Meanwhile, bigger amplitude produces lower Fe-Cr microalloying and smaller particle size and crystallite size.

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6. Referensi

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