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# The observation of resistivity change on the ultrasonic treated Fe-Cr ODS sinter alloy under magnetic field influence

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**Abstract.** About the observation of resistivity change on the ultrasonic treated Fe-Cr ODS sinter alloy under magnetic field influence. This paper reported about the observation of the resistivity change in the ultrasonic pre-treated Fe-Cr ODS sinter alloy under the influence of magnetic field at the Center for Science and Technology of Advanced Material, Nuclear Energy Agency of Indonesia. Fe-Cr ODS alloy were sinthesized by vacuum sintering of Fe- and Cr-powder dispersed $Y_2O_3$. However, before sintering the powder mixture was subjected to the irradiation process by ultrasonic for 50 hours at 20 kHz and then isostatic pressed up to 50.91 MPa to form a coin of 10 mm in diameter. LCR meassurement revealed the decreasing of resistivity about 3 times by increasing of applied magnetic field from 0 to 70 mT. In addition, VSM meassurement was performed on both as powder material and as sintered sample. The results showed increasing the magnetization with increasing magnetic field and the curve exhibits almost exact symmetry S-form with small hysterese indicating fast changing magnetization and demagnetization capability without energy loss. This opens strong speculations about the existence of magnetoresistant property of the material which is important for many application in field of sensors or electro magnetic valves.

## 1. Introduction

The ODS steel is an advanced material which is projected to be used for a system that is operated at high temperature such as for structural of advanced fission nuclear reactors and of fusion nuclear reactor [1]. The development of synthesis of ODS steel is in progress to find the adequate method and its characteristics. The ultrasonic method has been successefully to synthesize the Fe-Cr microalloy, following sintering in quartz capsule[2-4]. The Fe-Cr microalloy powders has bcc structure, homogenous, and no oxide as well as sintering product[2-4]. Besides, the ultrasonic method has the application for reducing the size particles that has effects on decreasing magnetoresistant value due to external magnetic field[5]. The ultrasonic method utilizes the mechanical efect of ultrasonic and cavitaion explosion that could has temperature of about 5000K and of thousand bar pressure[6]. In this experimetal work the ultrasonic method was used to synthesize the Fe-Cr ODS alloy following sintering in quartz capsule. The properties of product were characterized by using LCR and VSM.

## 2. Experiments

The material used to make an alloy is Fe powder with particle size about 3 µm, Cr with particle size about 23 µm, and $Y_2O_3$ with particle size < 50 nm and Toluene with 90% purity as media for ultrasonic irradiation treatment. The ratio composition in weight percent of powders mixture Fe:Cr: $Y_2O_3$ is 87: 12:1 respectively. The ultrasonically irradiation treatment of powders mixture was performed on the toluene solution in beaker glass at frequency of 20 kHz sonic vibracell amplitude for 50 hours. The characteristics of microstructure of treated ultrasonically powders was identified using Scanning Electron Microscopy (SEM) equipped with Energy Dispersive Spectroscopy (EDS) for micrograph and elemental composition, using X-Ray Diffraction (XRD) for the phase, and using Vibrating Sample Magnetometer (VSM) for measuring the magnetic properties. The compacting process of ultrasonically treated powders is done using isostatic pressed at at load of 50.91 MPa to form a coin of 10 mm in diameter. The coin was capsulated in quartz in vacumm condition. The sintering process was done in induction furnace at 1300 $^{o}$C for 1 hour, and then left to cool down to room temperature in the furnace. Characterization of the sintered Fe-Cr has been carried out using X-ray diffraction, VSM and LCR meter for resistivity as function of applied magnetic field.

## 3. Result and Discussion

XRD diffraction pattern of ultrasonically treated powders and its sintered are shown in Figure 1.

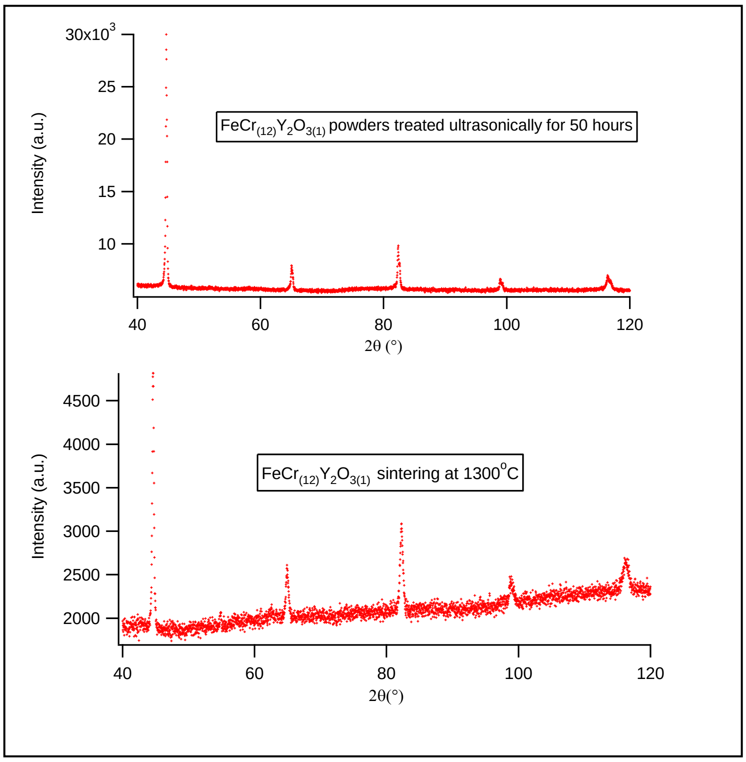


**Figure 1**. X-Ray diffraction intensity pattern of $FeCr_{(12)}Y_2O_{3(1)}$ powders treated ultrasonically for 50 h (a), and of sintered in quartz capsule at 1300 oC for 1 h. The both of peaks are consistent in similar position of bcc structure.

Figure 1 is a X-Ray diffraction intensity pattern of $FeCr_{(12)}Y_2O_{3(1)}$ powders treated ultrasonically for 50 h (a), and of sintered in quartz capsule at 1300 oC for 1 h. The both peak pattern of Figure 1 are similar with the bcc structure as found in earlier research[2,3]. Ultrasonic irradiations in toluene solution produce Fe-Cr microalloying as shown in Figure 2. As in Figure 2 the EDS of particles of area has composition of Fe = 98.26 % and Cr =1.74 %, and of spot has the composition of Fe = 3.44 % and Cr= 96.56 %. These particles are Fe dispersed Cr and Cr dispersed Fe.

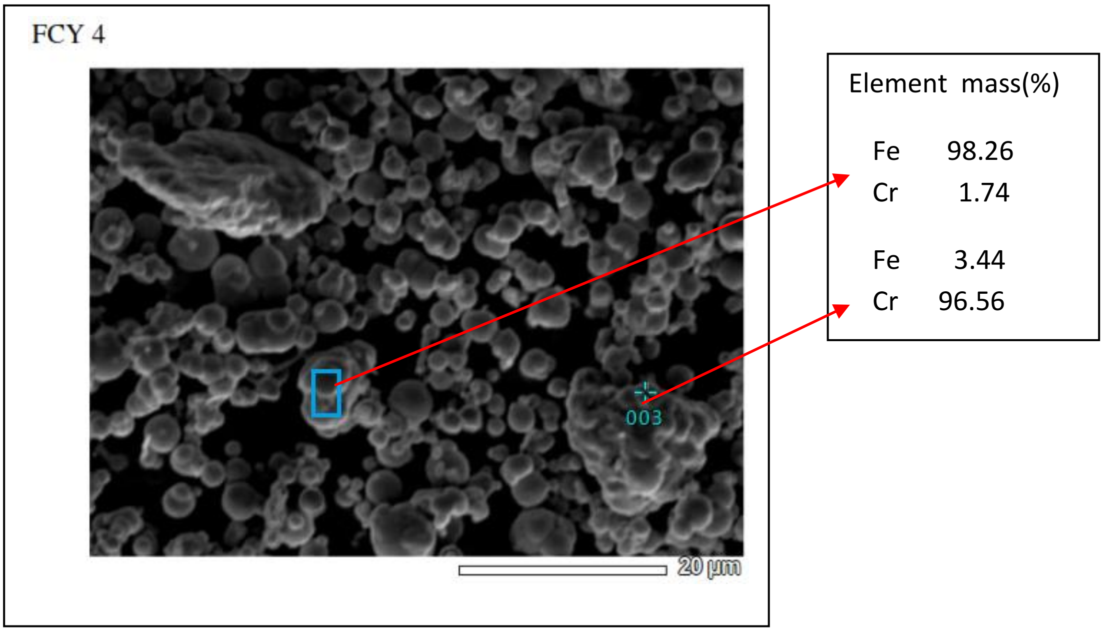


**Figure 2.** SEM Micrographs of the microstructure after irradiating for 50 hours at 20 khz of $FeCr_{(12)}Y_2O_{3(1)}$ powders. EDS result of powders of area were Fe dispersed Cr and point spot were Cr dispersed Fe.

The ultrasonic wave transmitted in toluene media can form stress and strain. These stress and strain can cause the particles to collide because the particles move to all directions[4] and also can cause cavitation nucleation[5,6]. The explotion of cavitation can produce high temperature and pressure that enabled Fe-Cr microalloying processes[2].

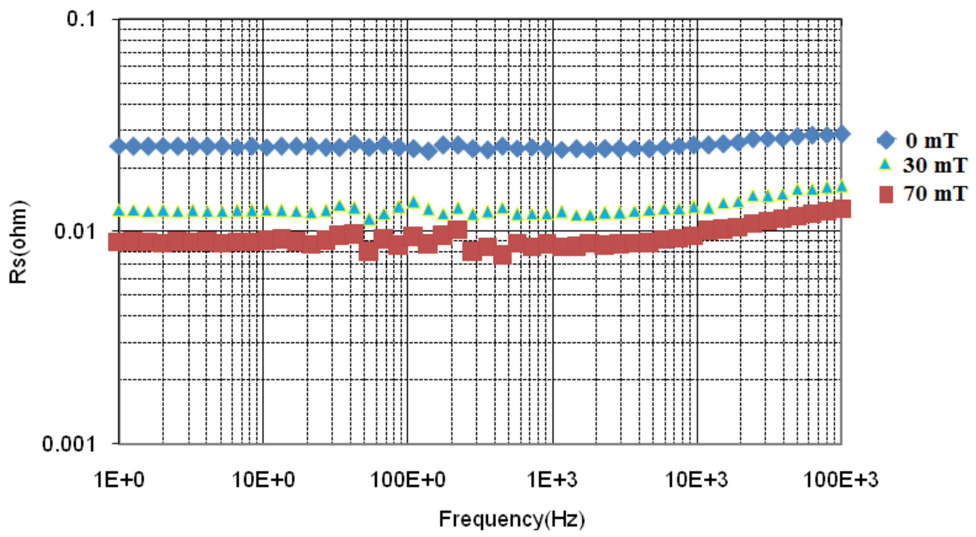


**Figure 3.** Resistivity data as function of applied magnetic field, blue (0mT), green (30 mT) and red (70 mT).

Figure 3 shown the Resistivity data of sintered $FeCr_{(12)}Y_2O_{3(1)}$ as function of applied magnetic field, blue (0mT), green (30 mT) and red (70 mT). The increasing of applied magntization up to 70mT decreased resistivity of 3 times. This phenomenon quite similar to the phenomenon of tunneling magnetoresistance in nanogranular system of Fe-$Al_2O_3$ as reported by S. Purwanto et al[7].

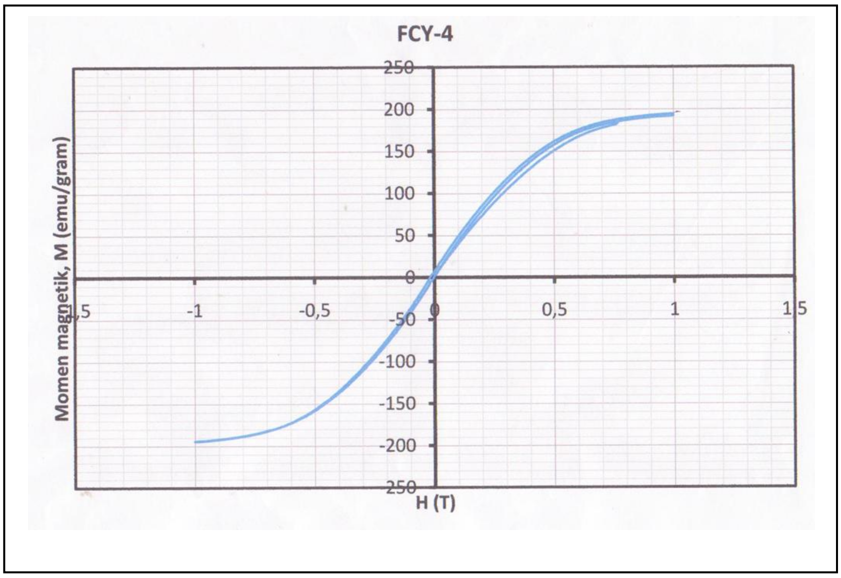


**Figure 4**. Magnetitation Diagram of $FeCr_{(12)}Y_2O_{3(1)}$ powder tretated ultrasonically for 50 hours.

In Figure 4 can be found that the $FeCr_{(12)}Y_2O_{3(1)}$ powder tretated ultrasonically for 50 hours showing high magnetic saturation and the absent of remanence and coercivity. The curve showed the almost exact symmetry S-form.

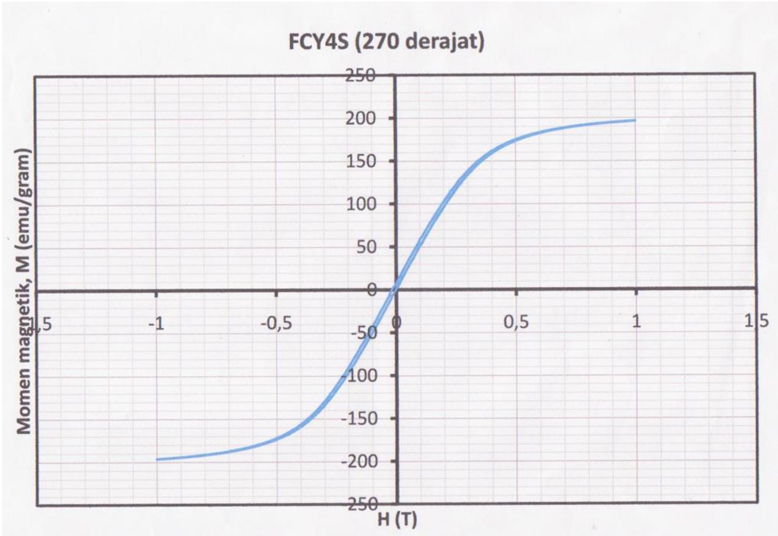


**Figure 5**. Graph magnetization of sintered $FeCr_{(12)}Y_2O_{3(1)}$ alloy taken in 270 $^oC$ angle.

Figure 5 shown the magnetization of sintered $FeCr_{(12)}Y_2O_{3(1)}$ alloy taken in 270 $^oC$ angle. For this magnetization angle, after sintered process the momen magnetic of Fe-Cr ODS irradiated ultrasonically was not changed.

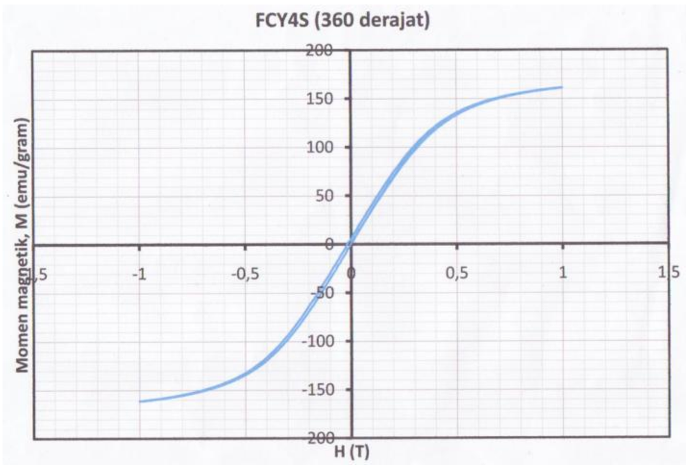


**Figure 6.** Graph magnetization of sintered $FeCr_{(12)}Y_2O_{3(1)}$ alloy taken in 360 $^oC$ angle.

In Figure 6 shown magnetization of sintered $FeCr_{(12)}Y_2O_{3(1)}$ alloy taken in 360 $^oC$ angle. The material is anisotropy as the momen magnetic is different in different angle. The structure and properties of Fe-based had been changed thorough adding Chromium, and also during milling the disordered magnetic phase, the reflectivity value of Fe-based powders (not adding Cr) was larger than FeSiAlCr powders[8]. Two kinds of atoms diffuse each other and solid solution reaction occurs on this interface[9]. A small magnetic moment or no moment alloy will be formed, and this results in reducing magnetic moment on magnetic layer. The existence of a few nanocrystalline phases results in a slight reduction of magnetization[10].

## 4. Conclusion

The Fe-Cr ODS alloy powders treated ultrasonically for 50 hours has the similar structure to the sintered in quartz capsule at 1300 $^{o}$C. The ultrasonic irradiating effects on Fe-Cr ODS alloy for 50 hours has decreasing of resistivity about 3 times by increasing of applied magnetic field from 0 to 70 mT. The results showed increasing the magnetization with increasing magnetic field and the curve exhibits almost exact symmetry S-form with small hysterese indicating fast changing magnetization and demagnetization capability without energy loss. This opens strong speculations about the existence of magnetoresistant property of the material which is important for many application in field of sensors or electro magnetic valves .

## 5. References

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