Study of magnetic properties of sintered alloy Fe-Cr ODS using VSM

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Abstract. In this experimental work Vibration Sample Magnetometer (VSM) was used to study the magnetization property of ultrasonic assisted sinter material ODS Fe-Cr containing dispersoid particles Y$_2$O$_3$. The results of magnetization diagram analysis showed an anisotropy with saturation values 150 and 200 emu/gr for the angles 270 and 360°, respectively. Although higher saturation level, the alloy exhibits exceptionally small hysteresis loop with very low remanence and coercivity. As powder Fe-Cr mixture before sintering has maximum saturation 200 emu/gr. Properties combination of anisotropy, the absent of hysteresis loop and high saturation value has been opened new research opportunity on application of Fe-Cr based ODS material in field of sensors and magnetic assisted electro mechanic parts such as transformers and motors.

1. Introduction
The search for a combination of materials to obtain a more powerful magnet is still a major topic in all parts of the world. Among them is the research conducted at the Center for Science and Technology of Advanced Materials (PSTBM) BATAN, in the framework of the investigation process of the formation of micro alloys, the Center has created a Fe-Cr ODS sintered alloy material by using ultrasonic preliminary process[1]. In publications that have also been presented in the same seminar, it was reported that this material has the properties of a magnetoresistance alloy. Measurements showed that the value of the materials resistance decreased with the effect of magnetic fields up to 3-fold. The purpose of this study was to determine the magnetic properties of sintered alloy material Fe-Cr ODS containing Y$_2$O$_3$ dispersed particles with measurement techniques using Vibrating Sample Magnetometer (VSM). VSM is a standard tool used for testing the magnetic properties of a material. In addition to the components and their functions VSM, observation characteristics Vibrating Sample Magnetometer (VSM) measurement results become the focus of discussion conducted in this paper.

2. Experimental Method
2.1. Material
The material used in this study is the ODS Fe-Cr alloy containing Y$_2$O$_3$. The chemical composition of the material is shown in table 1. Alloys are prepared as follows: first the powder starting materials Fe and Cr are prepared using the ultrasonic method for 50 hours at the frequency of 20kHz and 750 Watts power. Then the mixture was compacted by isostatic pressing method with a manual press
machine at a pressure of 6000 psi using molds with diameter of 10mm. Afterward, the pelletized solid sample is transferred into an ampoule made of a quartz glass material and then positioned inside the vacuum chamber. The ampoule and the sample is then sintered in an oven at 1000°C and 1300°C gradually and without interruption for one hour each. After the sintering process, the pelletized samples are taken out of the ampoule in a way to solve it, followed by polishing using sand paper to get rid of the passivation layer which may grow during the sintering process with residual air left in the ampoule in the vacuum process.

| No | material | contents [weight%] |
|----|----------|-------------------|
| 1  | Fe       | 87                |
| 2  | Cr       | 12                |
| 3  | Y$_2$O$_3$ | 1            |

2.2. VSM Components

All the VSM tool known today in principle has the same main components, namely as intended schematically in figure 1.

As shown in the figure can be broken down components of VSM following functions in sequence respectively as follows:

1. Head Generator is a place holder vibrator transmitted towards the sample in the sample holder or sample holder through a piezoelectric transducer.
2. The electromagnet or coil Hemholtz function generates a magnetic field to magnetize the sample then the process vibration transforms into electrical current that can be measured by the data processing system.
3. Pickup Coil transmit electrical signals from the sample into the amplifier. The signal will be transferred by the results of the induction coil pickup in the differential input to the lock-in amplifier. The signal from the pick-up coil is detected by a lock-in amplifier is noted as a function of magnetic field that allows us to get a hysteresis loop of the samples tested. For harmonic oscillation of the sample, the signal (e) induced in the pick-up coil is proportional to the amplitude of oscillation (K), the oscillation frequency of the sample (ω) and magnetic moment (m) of the samples tested.
4. The Hall Sensor is used to convert and transfer the magnetic field energy into voltage that will produce electric current. Hall sensors are also used to measure the flow without disrupting the flow.
of currents that exist on the conductor. The current measurement sensor connects hall with teslameter.

5. Sensor capacity serves to provide a signal proportional to the amplitude of oscillation of samples and supplies voltage to the electronic system which generates a reference signal. The next signal will be given to the reference input of the lock-in amplifier. Digital converter output will be sent to analog (DAC1out) and digital output (D1out) of the lock-in amplifier controls the current flowing through the electromagnet and show direction respectively.

Detailed information about the VSM has been published [2]–[10].

2.3. VSM Measurement
To prepare for VSM testing, a sample is cut to the size of 2x2x2 mm and then weighed. Weight of the sample was less than 10 mg, is the weight for optimum function of the sensor system in the VSM. In the next step of the process the samples were put into a small container and covered with wax to ensure that the position has not changed during the vibration measurement. After that the container together with the samples are inserted into the sample holder rod and fastened using a sealing tape, as shown in figure 2.

![Figure 2](image1.png)

**Figure 2.** The placement of the sample in the sample holder stem of the VSM test equipment.

The necessary steps in the testing procedure are outlined below and visually shown in figure 3;

![Figure 3](image2.png)

**Figure 3.** Working Principle vibrating sample magnetometer.

1. Put the sample into the prepared medium. Before running the VSM tool functions, the initial step that must be done is to place the sample holder between the poles of the magnetic field source.
2. Induction of the sample’s dipole moments. Samples that have been positioned will be conditioned by a uniform magnetic field. This happens because the magnetic induction performed by the edge of the VSM wall is magnetizing the pickups coil.
3. Measure the sample standard signal. Once the magnetic induction has completed the magnetization process, samples will show the signal in the form of vibrations with a sinusoidal motion in the medium pickup coil. This signal has the same frequency, where the sample will be proportional to the vibration amplitude and magnetic materials.

4. Output unit vibration magnetometer. Signals sent from the pickup coil system will be forwarded to the differential amplifier contained in the vibration unit. The output of the differential amplifier is then processed in an amplifier which receives a reference signal. And the end result of the process of identification of this signal will be given by the magnetometer in the form of a proportional DC signal which provides information magnetic moment of the sample being tested.

3. Results and Discussion

Figures 4 to 6 show the results of VSM testing in the form of charts showing magnetization as a function of magnetic field applied to the sample powder and solid samples taken from different directions with different angle of 90°. The results showed that the magnetization value of the material (as read off the chart), either in powder or solid state magnetism, is a very high value compared to the values in ferromagnetic materials or permanent magnets. The maximum value of the measured saturation is 200 emu/g at 1 Tesla magnetic field strength. Besides all the characteristics of a graph showing the results of magnetization with a slim hysteresis loop. It shows that all the samples do not have a remanence value or coercivity which indicates that the influence of even a small or weak external magnetic field will cause the magnetic properties of materials to immediately return to its original state. In other words, the material is inherent magnetic properties will not immediately disappear when the influence of the magnetic properties of the external magnetic field is removed. These properties are a very important quality to have for the components used in devices whose performance or operation depends upon the rapid changes in the magnetic field such as motors, transformers and relays.

As shown in figure 4, the powder sample has a saturation value of about 200 emu/g. This value is approximately equal to the results of measurements on solid samples and was taken at an angle of 270° (see figure 5). This shows that the state of the sample, whether as a powder or a sintered material has no effect on the value of magnetism, at least for one side of the sample. This could be an indication that the influence of magnetism is apparently caused by the ultrasonic process only, and not by the compaction process. However, because the ultrasonic process in this study was done on time, energy and the same frequency, so in this work, the authors cannot be certain what effects would occur when the ultrasonic treatment parameters were changed. In order to able to clarify these aspects, the authors would have to do further research.

![Figure 4](image-url). Graph magnetization VSM testing on samples of powder.
VSM measurement results in solid samples taken from different points of 90° or perpendicular to the previous measurement are displayed in figure 5, showing the value of saturation at 1 Tesla magnetic field recorded at 160 emu/g (figure 6). This value is lower, 40 emu/g, if measured at the angle of 270°. Analogous to what is commonly found in metallographic, this phenomenon shows the texture of magnetism, where the sample has a tendency toward a certain type of magnetism. Causes of what can be called a magnetic anisotropy have not been found in this study. There is a possible relation to the physical processes that occur during the mechanical pressing procedure. When pressed grains of powder materials change shape and become elongated in the horizontal direction and retracts in the axial direction, which metallographically speaking would be the cause of crystalline texture to arise in the material. However, the physical processes that occur in it is not the object of discussion in this study. It requires further research.

4. Conclusion
Based on the present study, it can be concluded the following: ODS sintered alloy Fe-Cr has a maximum saturation of 200 emu/g. The graph shows the magnetization of the material an isotropy on the angle of 90° with a sleek hysteresis loop shows instability in the magnetization of small magnetic field changes. This has opened up the possibility of new research linking objects between metallurgical and research-oriented magnetic sensor product in the form of materials or substances that work-based mechanical components magnetic.
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