Magnetic properties of oxide dispersion strengthened (ODS) ferritic steel

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The purpose of this study is to investigate the magnetic properties of oxide dispersion strengthened (ODS) ferritic steel. In this study, two samples of ODS ferritic steel powder were prepared by mechanical alloying method. The crystal structure and morphology of the samples were identified by X-ray Diffraction (XRD) analysis and examined by Field Emission Scanning Electron Microscopy (FESEM) respectively. The magnetic measurement including saturation magnetization (Ms), retentivity (Mr) and coercivity (Hc) of the samples were carried out at room temperature by using a Vibrating Sample Magnetometer (VSM). The magnetization curve of the samples (12Y and 14Y) approached the soft ferromagnetic behavior which is similar to its iron-based material due to the high content of iron (Fe) within the matrix. 12Y sample exhibit higher saturation magnetization, Ms value which is 295 emu/g due to the lower content of chromium (Cr) compared to 14Y sample which saturate at 162 emu/g.

Keywords: ODS ferritic steel; Soft ferromagnetic materials; Mechanical alloying; XRD; Magnetic properties.

1. INTRODUCTION

The attention of many researchers around the globe towards nanostructured materials is attributed to their mechanical, chemical, nuclear and physical properties as well as their potential application in many fields [1]. Oxide dispersion strengthened (ODS) ferritic steels is a one kind of this material and they are amongst the most promising candidates for large scale structural materials to be applied in next generation fission and fusion nuclear power reactors [2]. ODS

ferritic steels is an iron-based material and they are largely composed of magnetic elements which is iron. Like annealed iron which has been categorized as soft ferromagnetic materials [3], ODS ferritic steel can be manipulated under the influence of an external magnetic field to study the magnetic behavior of this material. According to Bramfitt (1998) [4], the iron-based material, ferritic stainless steels approached the properties of soft ferromagnetic materials which can be applied as electric solenoid cores with good corrosion resistance [5].
However, different chemical composition from the iron-based material along with the addition of oxide like Y_2O_3 or well known as dispersoids into the steel matrix could change the magnetic properties of this material. Basically, the addition of the dispersoids into the ferritic steel matrix is a strengthening technique to increase the thermal and nuclear stability [6,7]. For magnetic application, the properties of this material under the influence of external magnetic field are hard to find in the published literature. Therefore, the purpose of this present study is to investigate the magnetic properties of ODS ferritic steel at room temperature as this study is not much reported.

2. MATERIALS AND METHOD

High purity elemental powders (99% to 99.5%) of iron (Fe), chromium (Cr) and yttria (Y_2O_3) were weighed separately in a pure argon atmosphere and mixed according to the compositions in Table 1. The mixed powders were then sealed in a planetary ball mill Pulverisette 7 milling jar and the mechanical milling process was carried out at 350 rpm for 15 hours. The ball-to-powder weight ratio (BPR) is 5:1. After mechanical milling process, the milled powders were degassed at temperature of 400°C for 2 hours in a tube furnace filled with pure argon gas and the samples were allowed to cool inside. The crystal structure of the samples was identified by X-ray Diffraction (XRD) measurement (Bruker D8 Advance) with Cu Kα radiation and analyzed by using DIFFRAC.EVA V4.0 software. The morphology of the samples was examined by using a field emission scanning electron microscopy (FESEM) (Carl Zeiss Merlin). Prior to magnetic measurement, the samples were weighed for about 0.007 g and pressed inside a gelatine capsule following the sample preparation details given elsewhere [8]. The magnetic measurement including saturation magnetization (Ms), retentivity (Mr) and coercivity (Hc) of the samples was carried out by using a vibrating sample magnetometer (VSM) (Lakeshore 7404) in vacuum at room temperature for 500 sec experiment time.

Table 1 Compositions of ODS ferritic steel samples (wt%).

Sample	Designation	Fe	Cr	Y_2O_3
Fe-12Cr-0.3Y_2O_3	12Y	Balance	12	0.3
Fe-14Cr-0.3Y_2O_3	14Y	Balance	14	0.3

3. RESULTS AND DISCUSSION

3.1. Crystal structure and morphology of 12Y and 14Y ODS ferritic steel powders

The crystal structure of 15 h milled samples was identified by X-ray Diffraction (XRD) analysis. Fig. 1 shows the XRD patterns of 15 h milled 12Y and 14Y ODS ferritic steel powder samples. In comparison with JCPDS data card (PDF 03-065-4664), the main diffraction peaks (1,1,0 and 2,0,0) of both samples (12Y and 14Y) can be well indexed to iron (Fe) and chromium (Cr) which confirmed the complete solution of Cr into Fe lattice after 15 h milling time. The peak of Y_2O_3

is very low for both samples since the composition of this element is only 0.3 wt%. However, the XRD measurement still can detect the Y_2O_3 phase with the comparison of JCPDS data card of PDF 01-086-1326. The average crystallite size of 12Y and 14Y were calculated by using scherrer's equation (1) and were found to be ~13.9 nm and ~14.3 nm respectively as shown in Table 2. The broaden peak detected in the XRD patterns of both samples are consistent with the small crystallite size of the samples measured from XRD analysis by using scherrer's equation.

$$\tau = \frac{k\,\lambda}{B\,\mathrm{Cos}\,\theta} \qquad (1)$$

where τ is grain size, B is the full width at half maxima and λ is the wavelength of X-ray used which is 1.5406 Å and θ is the diffraction angle.

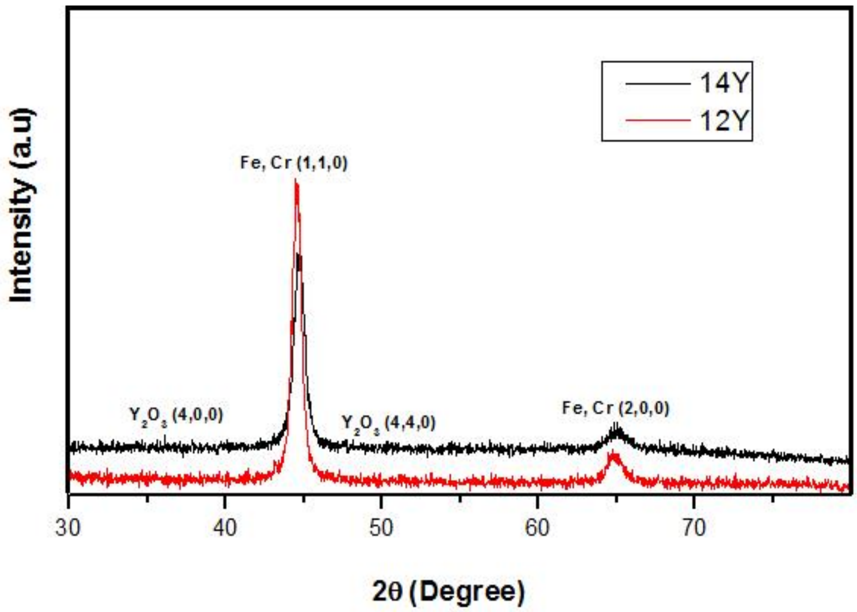

Figure 1 The crystal structure of 15 h milled 12Y and 14Y ODS ferritic steel powders

Table 2 Lattice parameters of 15 h milled 12Y and 14Y ODS ferritic steel powders.

Sample	Crystallite size (nm)	Unit cell parameters, a = b = c (Å)	Density (g/cm^3)
12Y	13.9	2.8664	7.767
14Y	14.3	2.8664	7.767

The morphology as well as the powder particles distributions of 15 h milled samples was examined by using field emission scanning electron microscopy (FESEM). Fig. 2 (a) and (b) shows the powder particles distributions of 15 h milled 12Y and 14Y ODS ferritic steel powders respectively which consist of flaky, angular and nearly spherical shapes powder particles with the average size of few hundreds nano. It is worth to remark that the particle size measured from XRD is slight different from the FESEM measurement because XRD measurement is done starting from the crystalline area that are reasonably diffracts by the X-ray waves coherently whereas FESEM measurement is done by taking into account the difference of noticeable grain

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boundaries [9].

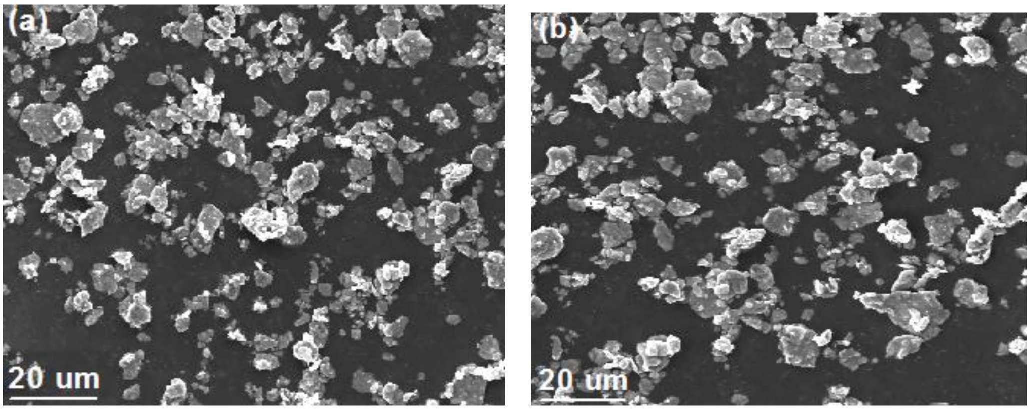
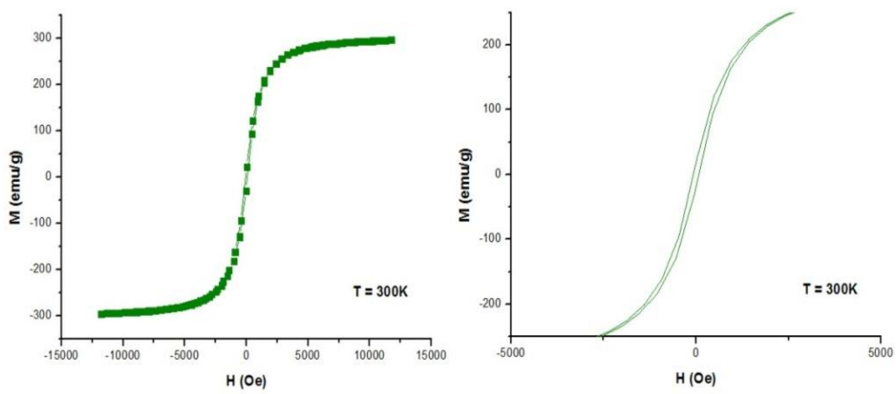


Figure 2 Powder particles distributions of 15 h milled ODS ferritic steel powders (a) 12Y and (b) 14Y

3.2 Magnetic measurement of 12Y and 14Y ODS ferritic steel powders

The magnetic measurement including saturation magnetization (Ms), retentivity (Mr) and coercivity (Hc) of 12Y and 14Y ODS ferritic steel powders was carried out by using a vibrating sample magnetometer (Lakeshore 7404) in vacuum at room temperature for 500 sec experiment time. Fig. 3 (a) and (b) shows the magnetization curve of 12Y and 14Y ODS ferritic steel powders recorded at room temperature respectively along with their clear hysteresis loop. The plot shows that both samples exhibit ferromagnetic behavior which is mainly generated by iron (Fe) as the main based as well as the matrix of this kind of material. The saturation magnetization (Ms), retentivity (Mr) and coercivity (Hc) value recorded for 12Y sample is 295 emu/g, 18.64 emu/g and 74.67 Oe respectively. While 14Y sample saturated at 162 emu/g with the retentivity and coercivity value of 10.99 emu/g and 82.61 Oe respectively. Since the magnetic coercivity value of both samples is small and the hysteresis loop is very narrow, ODS ferritic steel powders in this present study approaches the behavior of soft ferromagnetic materials which is similar to their iron-based material.

(a)



(b)

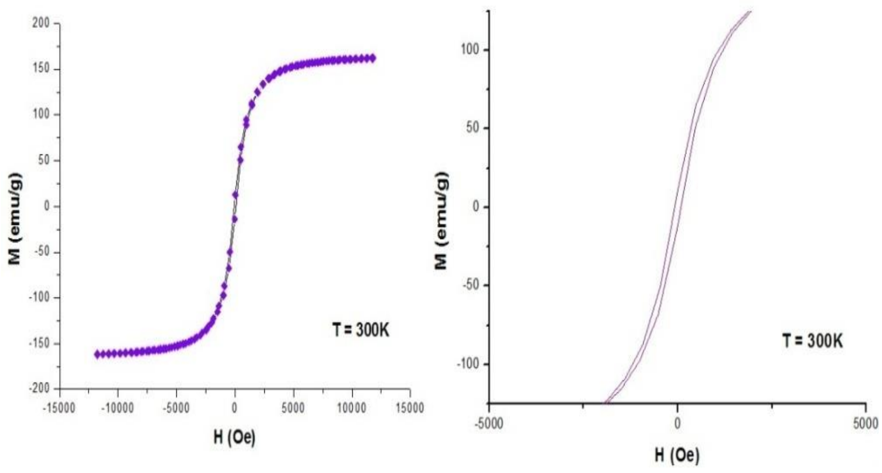


Figure 3 Magnetization curve of ODS ferritic steel powder along with their clear hysteresis loop (a) 12Y and (b) 14Y

Direct comparison with the saturation magnetization (Ms) value of other soft ferromagnetic iron-based materials is not possible since the samples of the same chemical composition were not available. Nevertheless, it is worth to note that the saturation magnetization value is closely related to the iron (Fe) content in the material. It is well known that an increase of iron content in the material will increase the saturation magnetization value, which will result in an increase of attractive force to the magnet. Thus, the saturation magnetization value of 12Y and 14Y ODS ferritic steel powders determined in this present study is consistent with the value reported by Flores et al. [10] which is 135 emu/g. The iron content in 12Y and 14Y samples in this study is in the range of 85% to 88% which is much higher than iron content in the PM2000 sample reported by Flores et al. [10] which is about 69%.

In comparison between both samples in this study, 12Y sample exhibit much higher saturation magnetization value compared to 14Y sample due to the lower chromium content which is 12 wt%. From the saturation magnetization value recorded for both samples and determined by using the law of approach to saturation, it is worth to remark that higher chromium content in ODS ferritic steel will decrease the saturation magnetization value as exhibited by 14Y sample. It also can be observed that the magnetic coercivity (Hc) value increase and the retentivity (Mr) decrease as the volume fraction of chromium (Cr) increase. Table 2 shows the summary of magnetic measurement results of 12Y and 14Y ODS ferritic steel powders determined in this present study. Overall, the results of magnetic measurement and magnetization curve of both samples recorded in this present study reveal that ODS ferritic steel approaches the behavior and properties of soft ferromagnetic materials which is similar to its iron-based material. The addition of small amount (0.3 wt%) of oxide like Y_2O_3 into the matrix also does not affect the soft ferromagnetic behavior exhibited by this material. Since the ferromagnetism behavior is strongly depends on the synthesis and processing techniques as well as the environmental conditions during the sample preparations [11], mechanical alloying method seems to be the best technique to successfully produced ODS ferritic steels that approach soft ferromagnetic behavior at room temperature.

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Table 3 Summary of magnetic measurement results of 12Y and 14Y ODS ferritic steel powders

Sample	Saturation magnetization, Ms (emu/g)	Retentivity, Mr (emu/g)	Coercivity, Hc (Oe)
12Y	295	18.64	74.67
14Y	162	10.99	82.61

4. CONCLUSIONS

In this study, the magnetic properties of ODS ferritic steel were investigated. The XRD analysis on the samples confirmed the complete solution of Cr into Fe lattice after 15 h milling time. The FESEM micrograph demonstrates the powder particles distributions of the samples which is flaky, angular and nearly spherical in shapes. The VSM has performed and record the magnetic measurement of the samples at room temperature along with the saturation magnetization (Ms), retentivity (Mr) and coercivity (Hc) values. The magnetization curve of ODS ferritic steel powder samples approach the soft ferromagnetic properties and behavior due to the high content of iron (Fe) which is similar to its iron-based material.

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