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Synthesis Oxide Dispersion Strengthening Stainless Steel doped with Nano Zirconia by Mechanical Alloying

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Abstract. The oxide dispersion strengthening stainless steel of Fe-11.5wt%Cr and Fe-11.5wt%Cr-1%ZrO_2 alloy by mechanical alloying method were synthesized by planetary ball milling. The methods employed for study were designing of Fe-11.5wt%Cr and Fe-11.5wt%Cr-1%ZrO_2 proportion of composition alloy which is plotted to *Schaffler* diagram to get ferritic/martensitic stainless steel. After MA the ODS powders were compaction with pressure 80kg/mm2 and followed by sintering at the temperature of 900,1000 and 1100^o C under high purity argon atmosphere for 1 hour. Characterization by XRD is used to examination phase present. Optical microscopy and SEM is used to get image microstructures. XRD analysis resulting the ferritic and martensitic is a major and minor phase respectively. There are not significant differences in the microstructure between Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO_2. An increase in the sintering temperature shift the microstructure from dendritic to equaxed. EDS examination showed that zirconia exit in the alloy Fe-11.5wt%Cr-1wt%ZrO_2.The addition of 1 % nano-zirconia (ZrO_2) into Fe-Cr alloy while milling process was resulted a higher Hardness Vickers Values rather than without zirconia addition. Average value of Hardness Vickers values was resulted 135.5 HV for Fe-11.5wt%Cr whereas 138.4 HV for Fe-11.5wt%Cr-1wt%ZrO_2.

1. Introduction

Current development of high-performance nuclear structural materials for breeding-blanket structural components, which will be exposed to high fluxes neutrons is one of the major challenges in future fusion reactors materials. The requirement of structural materials for the blanket components to a large degree dictates the design of the reactor systems. The selection of suitable structural materials is based on mechanical, thermophysical, corrosion and compatibility, low neutron inducedradioactivity, and resistance to radiation-induced damage phenomena like material hardening embrittlement [1].

Oxide-dispersion strengthened (ODS) stainless steels have been considered as promising high-temperature materials such as nuclear materials. High-chromium ferritic/martensitic steels are considered for the Generation IV nuclear reactors operated at elevated-temperature in-core applications. Basically, these steels contain 9–12% Cr to improve high temperature oxidation. [2,3]
Mechanical alloying (MA) is one of the methods to synthesize material uniformly by disperse nano-sized oxide particles such as yttria(Y2O3) in the metal matrix. This technique can be also employed to fabricate nano Fe alloys with the oxide dispersion [4,5,6].

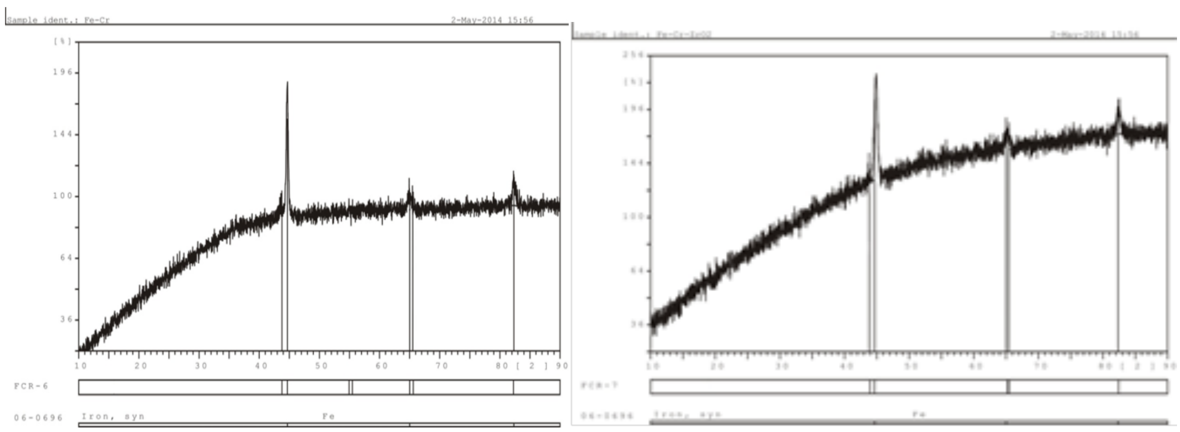
The purpose of this paper is to characterize the microstructural and mechanical properties (hardness) of ferritic/matensitic stainless steel of Fe-11.5wt%Cr and Fe-11.5wt%Cr-wt%ZrO2 as a candidate structural material for nuclear reactors application [7,8].

2. Experimental methods

The powder mixture with the nominal composition of Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO2 alloy was mechanically alloyed using Planetary Ball Milling (PBM) equipped with stainless steel vial for 2 hours. The milling balls were made of Zirconia with diameter of 2 mm. Ball-to-powder weight ratio was 10:1. The speed of rotating arm was 580 r.p.m. After MA the ODS powders were compaction with pressure 80kg/mm2 and followed to sintering at the temperature of 900,1000 and 1100^{o} C under high purity argon atmosphere for 1 hour. Micro hardness measurements were performed by using micro hardness tester HV 1000 equipped with a Vickers diamond pyramid and applying load of 0.98N for 15s. Each result is the average of at least 5 measurements. Characterization XRD by PHILIPS type PW 1835 device, using the Cu-K_ radiation with l=0.15406 nm. Optical microscopy and SEM is used to get image microstructures.

3. Result and discussions

X-ray diffraction patterns of the Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO2 alloy after sintering from planetary ball mill are shown in Figs. 1a and b, respectively. The peaks of elemental powder ZrO2, was not appeared in the diffraction pattern. The phase present is dominated by ferritic phase and martensitic is minor phase.



A b

Figure 1. a) Fe-11.5wt%Cr b) Fe-11.5wt%Cr-1wt%ZrO2 sinter at 11000C

Figure 2,3, and 4 shows representative optical and SEM-EDS micrographs of this Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO_2 stainless steel after sintering at 900^0C,1000^0C and 1100^0C. According to optical microscopy, there are not significant differences in the microstructure between Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO_2. After sintering at temperature 900^0C and 1000^0C. But an increase in the sintering temperature shift microstructure significantly at 1100^0C. The microstructure changed from dendritic to equated. Fig.4 showed that EDS examination depicted that zirconia exit in the alloy Fe-11.5wt%Cr-1wt%ZrO_2.

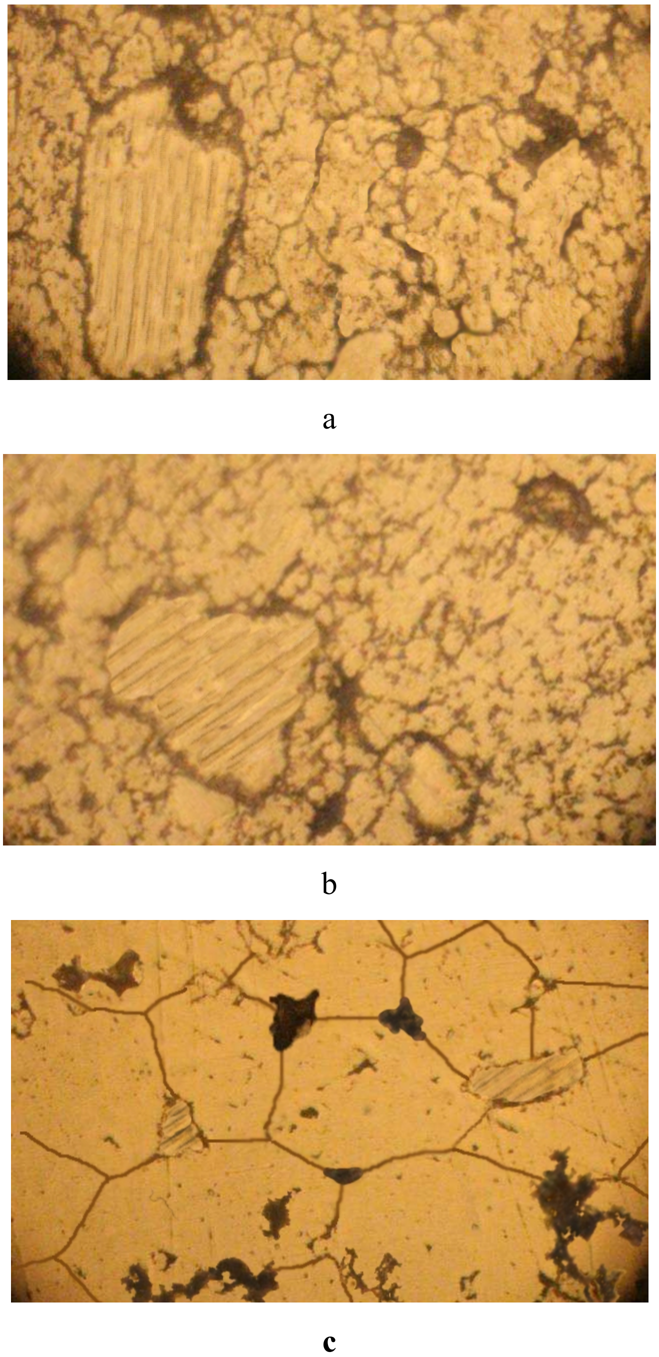
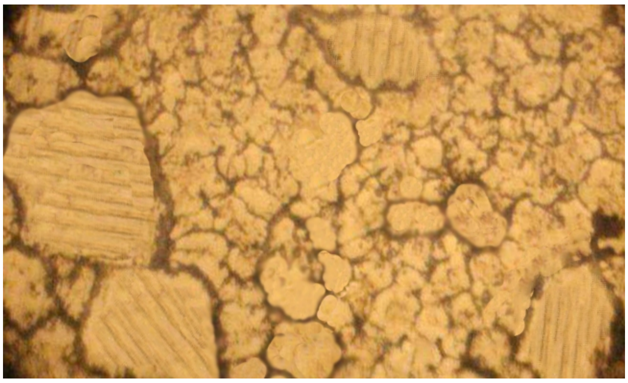
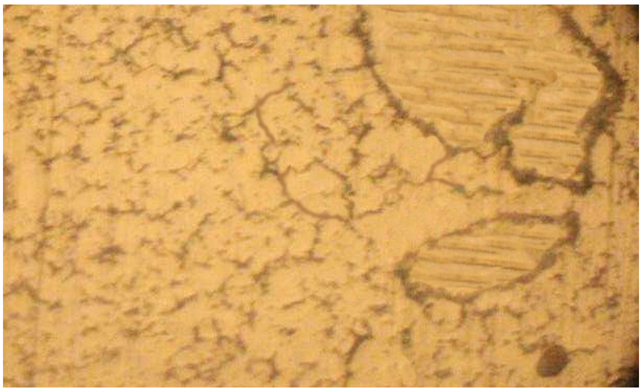


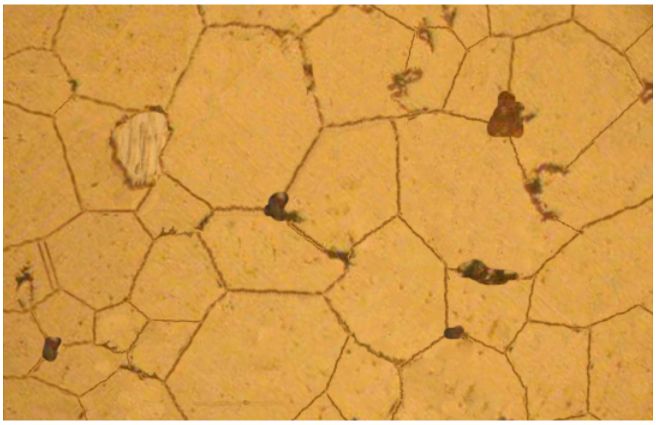
Figure 2. Microstructures of Fe-11.5wt%Cr sinter at a) 9000C b) 10000C c) 11000C, 25x



a



b



c

Figure 3. Microstructures of Fe-11.5wt%Cr-1wt%ZrO_2 sinter at a)900^0C b)1000^0C c)1100^0C, 25x

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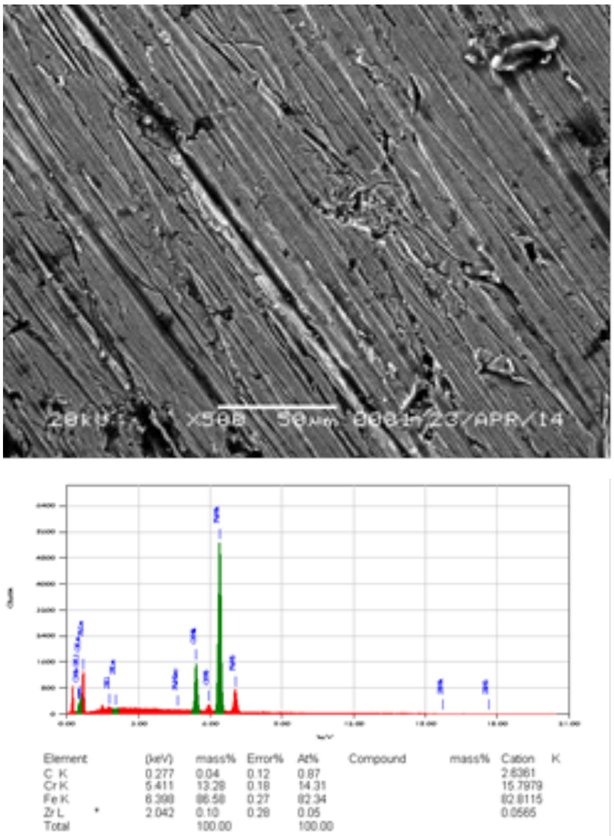

Figure 4. SEM-EDS Fe-11.5wt%Cr-1wt%ZrO2

Micro Vickers hardness measurements performed on Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO_2 have shown in figure 5. It was found that the micro hardness value of Fe-11.5wt%Cr-1wt%ZrO_2 highest compare with micro hardness value of Fe-11.5wt%Cr. Average value of Hardness Vickers values was resulted 135.5 HV for Fe-11.5wt%Cr whereas 138.4 HV for Fe-11.5wt%Cr-1%wtZrO_2. It can be concluded that the addition nano zirconia increase micro hardness Fe-11.5wt%Cr-1%wtZrO_2 Sintering temperature is also increasing hardness of Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO_2. Due to increasing density of the specimen.

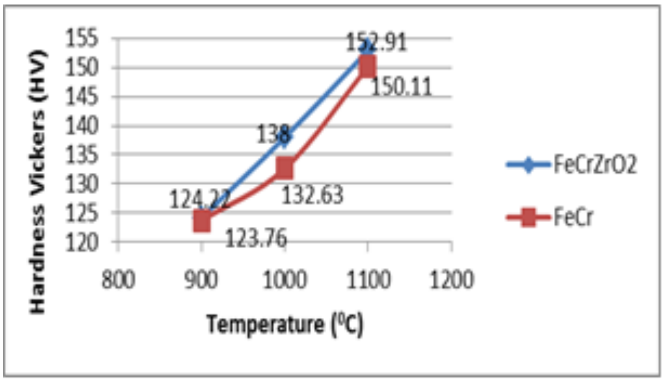

Figure 5. Micro Vickers hardness measurements

4. Conclusion

On the basis of the results the following conclusions can be as followed:

1. There are not significant differences in the microstructure between Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO2. EDS examination showed that zirconia exit in the alloy Fe-11.5wt%Cr-1wt%ZrO2
2. An increase in the sintering temperature shift the microstructure from dendritic to equaxed.
3. The XRD pattern was found that ferritic and martensitic were the major phase and minor phase, respectively.
4. It was found that the highest micro hardness value was achieved by addition nano zirconia. Average value of Hardness Vickers values was resulted 135.5 HV for Fe-11.5wt%Cr whereas 138.4 HV for Fe-11.5wt%Cr-1%wtZrO2. wtZrO2 Sintering temperature is also increasing hardness of Fe-11.5wt%Cr and Fe-11.5wt%Cr-1wt%ZrO2. Due to increasing density of the specimen.

Acknowledgments
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Reference

[1] 1. A. Méndez-Vilas and J. Díaz (Eds.), 2010 Microscopy: Science, Technology, Applications and Education, ©FORMATEX 2010 1811
[2] P. Fern´andez, A.M. Lancha, J. Lape˜na, R. Lindau, M. Rieth, M. Schirra, Creep strength of reduced activation ferritic/martensitic steel Eurofer'97 Fusion Engineering and Design 75–79 (2005) 1003
[3] E. Daum, K. Ehrlich, M. Schirra, Proccedings of the second milestone meeting on development of ferritic/martensitic steels for fusion technology, FZKA Report,FZKA, 1997, May, No. 5848.
[4] J. Lapen, M. Garcia-Mazarı´o, P. Ferna´ndez, A.M. Lancha ,Chemical segregation behaviour under thermal ageing of the low activation F–82H modified steel, J. Nucl.Mater. , 662, (2000), 283–287
[5] Benjamin J. S**.**, Mechanical Alloying, *Scientific American, 234, (1976), 40-8.*
[6] Mukhopadhyay D. K., Froes F. H., Gelles D.S. Development of oxide dispersion strengthened ferritic steels for fusion, *Journal of Nuclear Materials*, Vol.1209, (1998) 258-263.
[7] Ohtsuka S., et al**.** , Nano-structure control in ODS martensitic steels by means of selecting titanium and oxygen contents, *Journal of Physics and Chemistry of Solids*, Vol. 66,(2005)571-575.
[8] Patil U., et al., An unusual phase transformation during mechanical alloying of an Fe-based bulk metallic, *Journal of Alloy and Compounds*, Vol. 389, (2005)121-126.
[9] Suryanarayana , Mechanical alloying and milling, *Progress in Mat. Science*, Vol. 46,(2001) 1-184.