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Synthesize And Investigate The Austenitic Nanostructural Propertise

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Abstract

The austenitic stainless steel (SS) has been used as a material for building ultra high vacuum (UHV) chamber. Recently significant efforts have been concerned a comparing the standard cleaned for ultra high vacuum, passivity stainless. In this work Austenitic nanocomposites were synthesize by sol- gel method. The morphologies and topographies of the samples have been studied with using Scanning Electron Microscopy (SEM) and X-Ray diffraction (XRD) Techniques. The principal aim is the Nanostructural evolution of these samples. The obtained results indicate that adding 10.8 gr Ni particles into SS matrix,modified SS device mechanical structures.

1. Introduction

Nanoscale materials exhibit unique chemical and physical properties [1-5]. In particular, nanocomposites composed of metal oxide nanoparticles embedded in silica matrices show magnetic, electric and catalytic properties anel strongly depend on particle size [4-8]. To grow an ultra thin oxide film we need a UHV chamber which can keep low pressure down to 10^{-14} Torr. The current UHV chamber can not keep such a low pressure due to leakage and some unwanted bonds on the SS structures. Henceforth the vacuum chamber should stand its stability to prevent corrosion. One of the problems for UHV chamber designers is the practical difficulty of detecting of the navel properties of nano crystalline austenitic stainless steel.

This type of steel used in nano-electronics industries, optoelectronic, covered containers vacuum, food equipment and household applications and building [3]. We have thus tried to synthesize nano Nickel particle in stainless steel (SS) content to fabricate more stable SS-UHV chamber by sol-gel method. The sol- gel process is commonly applied to synthesis such SS composite owing to its several advantages such as low temperature processing and the ability to prepare materials in various shapes, compared with the conventional preparation procedures of glass and ceramics.

This method is one of the most usual, well-studied, available, fast and inexpensive technique. The condition of preparation during the sol-gel process plays a great role in formation of uniform metal nanostruture [5, 9]. In this paper the XRD, SEM, EDX and X-powder techniques have been used to characterization of this nanocomposite involving Ni nanocrystallines.

2. Experimental procedure

Nanocomposite austenitic SS synthesized by the sol- gel method. The materials used for the synthesis were:

16 gr $Fe(NO_3)_3$, 9H2O (Merck), $Ni(NO_3)_2$, 6H2O (Merck), $Zn(NO_3)_2$, $6H_2O$ (Merck), ethylene glycol (p.a.), 15 mm ethanol (C_2H_5OH) and 25 mm Tetra Ethyl Ortho Silicate(TEOS) (Fluka,98%).

The molar ratio of Fe nitrate to Zn and Ni nitrates were 0.25 and 1.4 respectively. The metallic nitrates, weighed to the designed stoichiometric ratio, have been dissolved in ethyleneglycol and ethanol. The obtained solution has been slowly added in drops, while stirring, to the ethanol TEOS solution. Ethanol has been added in order to increase the miscibility of the two solutions.

The homogenous, clear solutions obtained in this syntheses were subordinate to stirring at room temperature and air atmosphere to gellify. After 24h stirring, the sol was transmuted to gel. The obtained gels have been dried in the drying oven at 80 ºC, for 15 minutes. After drying and milling, the resulted powders have been thermally treated at different temperatures and than characterized.

3. Results and discussion

To synthesize the $Ni_{1.4}Zn_{0.25}Fe_2O_4$ austenitic in SiO_2 matrix the reactions obey below relation:

$$2Fe(NO_3)_3+1.4Ni(NO_3)_2+0.25Zn(NO_3)_2+ 2C2H_4(OH)_2+xH_2O \rightarrow Fe_2Ni_{1.4}Zn_{0.25}(OH)_4(C_2H_2O_4). xH_2O + 4NO + 4HNO_3$$

The samples were analyzed by using X-Ray diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques. Fig. 1, 2 and 3 show XRD patterns of austenitic nanocompositesn obtained by sol- gel method. As shown in figure 1, the sample prepared at 300 °C is amorphous.

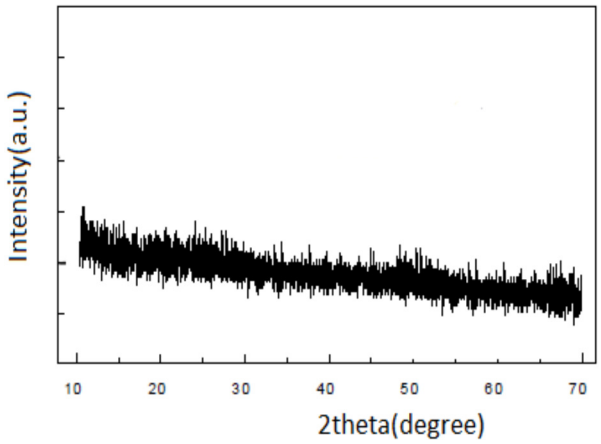

Figure 1. XRD patterns of samples, calcined at 300 °C

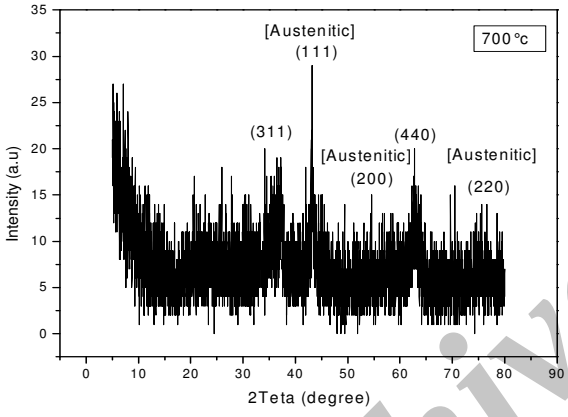

Fig. 2. XRD patterns of samples , calcined at 700 ° C

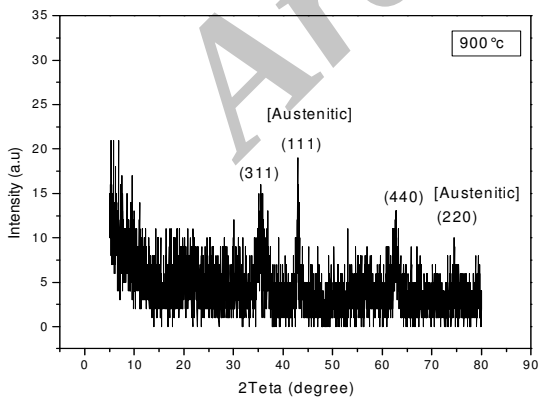

Fig. 3. XRD patterns of samples , calcined at 900 °C

In Fig. 2 it let to crystallization structure at 700 °C. The five dominated peaks labelled with miller indexes (See figure 2) exhibit surface centered cubic phases.

As Shown in Fig. 2, peaks located at 35°, 43°, 50°, 63° and 75° attributed to the (311), (111), (200), (440) and (220) diffraction plans of the face center cubic (FCC).

Fig. 3 displays the XRD pattern at 900 °C in where intensities and values are less than those low temperatures.

The size of nanoparticles was estimated by X-powder software (based on Debye-Scherrer equation) and given on the figures 4 and 5.

$$D = \frac{k\lambda}{\beta \cos\theta}$$ Debye–Scherrer equation

Where D is the crystallite size of nanoparticle K is a constant (0.94), λ is the wavelength of X-ray (($Cu_{k\alpha}$ = 1.5406 Å), β is the true half- peak width, and is the half diffraction angle of the centered of the peak in degree.

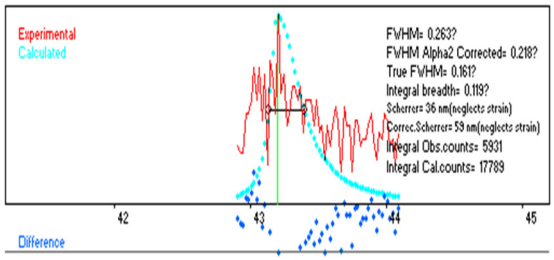

Fig. 4. X-Powder Technique of sample calcined at 700°c

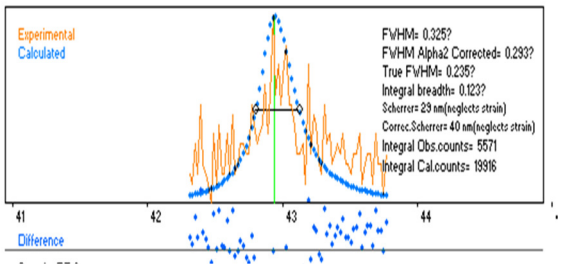

Fig. 5. X-Powder Technique of sample calcined at 900°C

It is clear there are different values of nanoparticle size with to different methods. Its calculated issues exhibit that the nanoparticles size was decreased from 59 nm to 40 nm while temperature was raising from 700 ˚C to 900 ˚C for (111) phase, located at 43° as shown in histogram of Fig. (4,5). Because, by reducing the size of the Nanoparticle, mechanical strength increases.

Fig. (6, 7) represented the SEM images of samples austenitic synthesize at 700 °C , 900°Ccalcinated temperature. The reason is that scherrer equation can be only used for spherical particle.

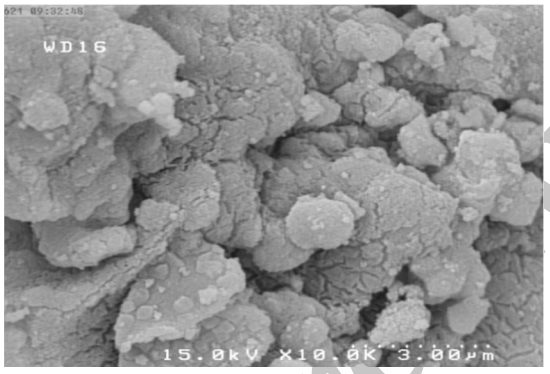

Fig. 6. SEM Images of sample calcined at 700°C

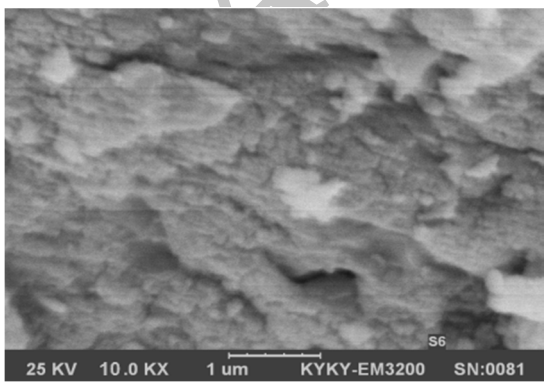

Fig. 7. SEM Images of sample calcined at 900°C

The obtained results indicate that a more mechanical stable ultra high vacuum chamber structure which can reduce leakage current and keep its structure may be produced for samples synthesize at high (> 800 °C) temperature.

Comparative samples show, the temperature increases the rate of cavities and cracks are reduced and have more pairs, which may indicate leakage flow into the chamber is reduced.

4. Conclusion

The prcent procedures at high calcinated temperature can be used for the future of ultra high vacuum chamber productions due to reduction of leakage current and more stable structure.

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