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Fabrication of in situ Al_2O_3 reinforced nanostructure 304 stainless steel matrix composite by self-propagating high temperature synthesis process

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ABSTRACT

304 austenitic stainless steel reinforced by Al_2O_3 particles was prepared by microwave assisted self-propagating high temperature synthesis process using the $Fe_2O_3Cr_2O_3NiOAlFe$ reaction system. Furthermore, effects of mechanical activation of the reactants and the addition of 21.2 wt.% extra Al to the chemical composition of the reactants on the chemical composition of the produced stainless steel was investigated. Atomic absorption spectroscopy analysis results indicated that by the addition of extra Al to the reactant mixture and using 30 minute mechanical activation, stainless steel containing 17.27 wt.% Cr and 7.73 wt.% Ni could be produced with its chemical composition very close to the chemical composition of 304 stainless steel. X-ray diffraction analysis showed that the stainless steel contains nanostructured austenite and ferrite phases. Also microstructural characterizations indicated that there is a uniform distribution of black particles in the steel matrix. Energy dispersive spectroscopy analysis showed that these particles are composed of Al and O elements while the matrix contains Fe, Cr and Ni elements. The presence of Al_2O_3 particles and nanostructure matrix improved the hardness and therefore the wear properties of the composite in comparison with the wrought 304 stainless steel plate.

1. Introduction

Stainless steels are a group of materials, which usually are used for corrosion resistance applications. Austenitic stainless steels are the most common type of stainless steels. Good corrosion resistance and welding characteristics, high formability and nonmagnetic nature due to the presence of fully austenitic microstructure are the most common properties of these alloys [1–5]. 304 austenitic stainless steel is the most common type in the AISI 300 series with chemical composition, including 18 wt.% Cr, 8 wt.% Ni, and balance Fe. There are several methods for fabricating stainless steels [6]. From energy-saving viewpoint, combustion synthesis (CS) process is one of the most attractive techniques for preparing these materials. In this process, an exothermic reaction is used for fabricating the final product. The combustion synthesis process usually is divided to volume combustion (VC) synthesis and self-propagating high temperature synthesis (SHS). In the VC, the entire sample is heated uniformly in the furnace. In this condition the reaction starts in all parts of the sample simultaneously while during SHS, the sample is heated locally by an external heat source like spark, tungsten filament or microwave. Once the reaction starts, the heat liberated from the reaction provides the necessary heat for its propagation along the entire sample [7].

Fabrication of stainless steels by SHS process can have some difficulties due to the presence of different alloying elements in the composition of the alloy. Gowtam and co-workers [8] produced Fe–Mn austenitic stainless steel reinforced by TiC particles via SHS process. They used aluminothermic reaction of Fe_2O_3, MnO and TiO_2 in the presence of carbon for the recovery of the alloying elements. Their results indicated that process parameters, including green composition, blending sequence and average particle size of Al had considerable influence on the Mn and TiC recovery and the final microstructure of the composite.

Xi and co-workers [9] produced stainless steel layer in the inner surface of a low-carbon steel pipe via loading aluminothermic reaction of Fe_2O_3, Cr_2O_3, CrO_3 and NiO into it and centrifugal SHS process. They discussed about the possibility of crack formation in the stainless steel layer, and its avoiding ways. Their results showed that the stainless steel contains 12–14% Cr, 1416% Ni and 70–74% Fe. Furthermore, microstructure analysis indicated that some ferrite layers were distributed in the austenite grain boundaries along with AlNi precipitates into the ferrite.

But, up to now there is no report on the fabrication of 304 stainless steel by SHS process using the $Fe_2O_3Cr_2O_3NiOAlFe$ reaction system. This study presents a novel process for fabricating 304 stainless steel using aluminothermic reactions of Fe_2O_3, Cr_2O_3, NiO, Al and also some

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Table 1
Chemical composition of powders and process parameters in the different series.

Sample name	Fe (wt.%)	Fe_2O_3 (wt.%)	Cr_2O_3 (wt. %)	NiO (wt. %)	Al (wt.%)	Mixing conditions	Chemical composition
A	43.14	15.85	19.52	7.43	14.06	Unmilled	Stoichiometric mixture
B	41.89	15.39	18.95	7.21	16.55	Unmilled	21.2 wt.% extra Al
C	43.14	15.85	19.52	7.43	14.06	Milled for 30 min	Stoichiometric mixture
D	41.89	15.39	18.95	7.21	16.55	Milled for 30 min	21.2 wt.% extra Al

Fe as diluent agent using microwave assisted self-propagating high temperature synthesis processes.

2. Experimental works

In this study Al (99.5%, 20 μm), Fe_2O_3 (99%, 30 μm), Fe (96%, 100 μm), Cr_2O_3 (99.8%, 30 μm) and NiO (99.7%, 45 μm) powders were used as raw materials. The chemical composition of 304 austenitic stainless usually contains 8 wt.% Ni, 18 wt.% Cr and balance Fe. The presence of Ni and Cr alloying elements leads to stabilization of austenite at room temperature and increases the corrosion resistance of this type of steels. So, in order to fabricate 304 stainless steel, the mixture of the raw materials should be selected in such a way that the weight percent of the Ni and Cr alloying elements in the composition of the final product be 8 wt.% and 18 wt.%, respectively. Therefore, the following reaction was considered for synthesis of 304 stainless steel by combustion synthesis process.

$$x\,\mathrm{Fe} + (0.66-x/2)\,\mathrm{Fe_2O_3} + 0.175\mathrm{Cr_2O_3} + 0.135\mathrm{NiO} + (1.760-x)\mathrm{Al} = 1.32\,\mathrm{Fe} + 0.35\mathrm{Cr} + 0.135\mathrm{Ni} + (0.88-x/2)\mathrm{Al_2O_3}. \quad (1)$$

In order to control the heat liberated from the reaction and fabrication of a nanostructure stainless steel, some of the Fe_2O_3 was replaced by Fe diluent agent. So, in reaction (1), x denotes the mole of the diluent agent added to reaction (1) which is 1.05 mol in the present work. The reason for the selection of this value is presented in the next section. Also, with the addition of Fe, the volume fraction of Al_2O_3 in the final product can be controlled. Therefore, in the present research four different samples were selected for the fabrication of 304 stainless steel based on reaction (1) and investigating the effects of the mechanical milling of the powder mixtures and the addition of 21.2 wt.% excess Al to the reaction on the element recovery from their oxides during synthesis. Mechanical milling of the powder mixtures was performed by a planetary ball mill (M200, Iran) using some stainless steel balls with a ball to powder ratio of 10/1 and a rotation speed of 200 RPM for 30 min. Table 1 shows the chemical compositions of powder mixtures in different samples.

It should be noted that during mechanical milling some ethanol droplets were used as process control agent (PCA) to reduce the agglomeration of powders. After mixing, two grams from each mixture was uniaxially pressed and cylindrical pellet with a diameter of 12.8 mm and 70% of theoretical density was produced. Then the pellets were placed in a microwave apparatus (Butan Co., Iran) operating at 2.45 GHz in ambient temperature and pressure. To facilitate the ignition of the synthesis reaction and avoiding preheating of the samples, a small piece of copper was placed on top of the pellets.

After the synthesis process, a slag layer covered the main product of the synthesis. In order to separate this layer, pellets were cold rolled. Then, the atomic absorption spectroscopy (AAS) apparatus (Shimadzu-AA-760) was used for measurement of Fe, Cr, and Ni weight percent in the products. Also, X-ray diffraction with Cu Kα radiation (PC 1800) was used for phase analysis of the samples. The microstructures of the samples were analyzed by an optical and scanning electron microscope (SEM LEO 1450 VP, Germany) equipped with energy dispersive spectroscopy (EDS Oxford INCA 7353). Dry pin on disk sliding wear test of the composite was performed on the 180 emery paper using 5 and 10N normal loads for 600m sliding distance. The weight loss of the samples was measured by a precision digital balance at each 50 m interval. To compare the wear properties of the composite, the wear test was performed on a wrought 304 stainless steel plate (18.15 wt.% Cr, 8.20 wt.% Ni, 1.75 wt.% Mn, 0.11 wt.%Mo, 0.70 wt.%Cu, 0.04 wt.% C and balance Fe) in the same conditions. Also, the hardness of the composite was measured by a Vickers hardness tester.

2.1. Thermodynamic considerations

As stated before, the main purpose of the present study was fabricating a nanostructured 304 stainless steel reinforced with alumina particles. Clearly during synthesis processes, as the temperature increases, the growth process of the products will happen more easily. So, to fabricate a nanostructure stainless steel, the temperature of the combustion synthesis process should be decreased as low as possible. This can be performed by the addition of a diluent agent to the reaction. So, as stated in the Experimental works section, in the present study some of Fe_2O_3 was replaced by Fe as a diluent agent to decrease the combustion synthesis process temperature and formation of nanostructure stainless steel. On the other hand, if the synthesis temperature decreases considerably, the stable propagation of the combustion wave may not be performed. The adiabatic temperature (T_{ad}) which is the highest temperature that a reaction system can reach in adiabatic conditions is a criterion which usually is used to find the theoretical stability instability regions of a combustion synthesis system. In a reaction system, the adiabatic temperature can be calculated using the following formula [7,9,10]:

$$-\Delta H^0_{298} = \int_{298}^{T_{tr}} nC_p(products)dT + n\Delta H_{tr} + \int_{T_{tr}}^{T_{ad}} nC_p(products)dT \quad (2)$$

where ΔH^0_{298} is the difference between formation molar enthalpy of products and raw materials at 298 K, n is the mole of each product and C_p is the molar heat capacity. If there is any phase change in products during heating, it should be considered in the calculations. So, in the previous formula, T_{tr} is the transformation temperature and ΔH_{tr} is the molar enthalpy of transformation. Also, if a material is added to the reaction as diluents, its heat capacity should be added to the product's heat capacity [7]. According to the experimental criterion of Merzhanov [11–13], in an exothermic reaction if the calculated

Table 2
Chemical compositions of the products measured by atomic absorption spectroscopy analysis.

Sample name	A			B			C			D		
Alloying elements	Fe	Cr	Ni	Fe	Cr	Ni	Fe	Cr	Ni	Fe	Cr	Ni
Mean wt.% in the composite	76.17	6.81	5.15	77.38	8.87	6.06	72.1	13.83	6.94	70.87	16.33	7.31
Mean wt.% in the composite matrix	86.42	7.73	5.84	83.82	9.61	6.56	77.63	14.89	7.47	74.98	17.27	7.73