J MATER RES TECHNOL. 2017;xxx(xx):xxx–xxx

Available online at www.sciencedirect.com

jmr&t Journal of Materials Research and Technology

www.jmrt.com.br

Original Article

Optimization of process parameters for spark plasma sintering of nano structured SAF 2205 composite

Samuel Ranti Oke[a,b,*], *Oladeji Oluremi Ige*[a,c], *Oluwasegun Eso Falodun*[a], *Babatunde Abiodun Obadele*[a], *Mxolisi Brendon Shongwe*[d], *Peter Apata Olubambi*[a]

[a] *Center for NanoEngineering and Tribocorrosion, School of Mining, Metallurgy and Chemical Engineering, University of Johannesburg, Doornfontein Campus, 2028 Johannesburg, South Africa*

[b] *Department of Metallurgical and Materials Engineering, Federal University of Technology Akure, Ondo State, Nigeria*

[c] *Department of Materials Science and Engineering, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria*

[d] *Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa*

ARTICLE INFO

Article history:
Received 2 February 2017
Accepted 22 March 2017
Available online xxx

Keywords:
Spark plasma sintering
Duplex stainless steel (SAF 2205)
Titanium nitride (TiN)
Microstructure
Density
Hardness

ABSTRACT

This research optimized spark plasma sintering (SPS) process parameters in terms of sintering temperature, holding time and heating rate for the development of a nano-structured duplex stainless steel (SAF 2205 grade) reinforced with titanium nitride (TiN). The mixed powders were sintered using an automated spark plasma sintering machine (model HHPD-25, FCT GmbH, Germany). Characterization was performed using X-ray diffraction and scanning electron microscopy. Density and hardness of the composites were investigated. The XRD result showed the formation of $FeN_{0.068}$. SEM/EDS revealed the presence of nano ranged particles of TiN segregated at the grain boundaries of the duplex matrix. A decrease in hardness and densification was observed when sintering temperature and heating rate were 1200 °C and 150 °C/min respectively. The optimum properties were obtained in composites sintered at 1150 °C for 15 min and 100 °C/min. The composite grades irrespective of the process parameters exhibited similar shrinkage behavior, which is characterized by three distinctive peaks, which is an indication of good densification phenomena.

1. Introduction

Duplex stainless steels (DSS) is a prominent member of the stainless steel family, which combines the properties of both austenitic and ferritic classes of stainless steel because of the near balance of austenite and ferrite phases present in its microstructure [1–5]. They have been successfully utilized in aerospace, chemical, power generation and biomedical industries and other demanding engineering applications due to

* *Corresponding author.*
E-mail: sroke@futa.edu.ng (S.R. Oke).

http://dx.doi.org/10.1016/j.jmrt.2017.03.004

Please cite this article in press as: Oke SR, et al. Optimization of process parameters for spark plasma sintering of nano structured SAF 2205 composite. J Mater Res Technol. 2017. http://dx.doi.org/10.1016/j.jmrt.2017.03.004

Table 1 – Chemical composition of SAF 2205 and TiN (wt%).

Elements	C	Si	Mn	P	S	Cr	Ni	Mo	N	Ti	Fe
SAF 2205	≤0.03	≤1.0	≤2.0	≤0.03	≤0.015	22	5	3.2	0.18	–	Bal
TiN	0.03	<0.003	–	–	–	–	<0.001	–	21.91	77.83	<0.001

possession of attractive properties such as high corrosion resistance, good mechanical strength and ductility, abrasion resistance, erosion resistance and a very good weldability [6–9].

Conventional DSS alloys suffer considerable loss in wear and mechanical strength at high temperatures. Such limitations in mechanical properties limit their use in structural applications in many industrial sectors [10,11]. One promising approach to improve the elevated temperature properties as well as the mechanical, wear and oxidation properties of stainless steels is by reinforcing it with ceramic dispersoids [12].

The dispersion strengthened steels have attracted considerable attention due to their ease of fabrication, lower costs, and attractive physical and mechanical properties. Traditionally, reinforced steels have been produced by several processing routes such as powder metallurgy, conventional casting and reactive sintering techniques [13]. All these techniques are based on the addition of ceramic reinforcements to the steel matrix, which may be in molten or powder form [14,15]. Among these fabrication techniques for synthesizing the composites, spark plasma sintering (SPS) has attracted much attention, because of its low energy consumption and very short sintering time [16,17].

Spark plasma sintering (SPS) is a versatile technique used to rapidly fabricate a number of materials including metals [18], ceramics [19,20] and composites [21]. In the SPS method, starting powders are placed in a graphite die, and a uniaxial pressure is applied during sintering. The heating is accomplished by spark discharges in voids between the particles. Due to these discharges, the particle surface is activated and purified, and a self-heating phenomenon is generated between the particles. As a result, heat-transfer and mass-transfer can be completed instantaneously. Therefore, SPS technique can be used for sintering ceramic particulates reinforced composite quickly to its full density at relatively low temperature [22].

Several works have been reported for the SPS of stainless steel based composites [13,14,21,22] but nano particle dispersion strengthening of duplex stainless steels has received little attention from researchers. Also, the challenge to obtain fully dense or near-full dense composites and avoid grain growth with nano particles is of great importance and could be carefully resolved by appropriate selection of sintering process parameters to meet demanded mechanical properties and wear resistance [23,24]. However, it is imperative to state that this article presents the first set of data for our research which seeks to develop nano structured duplex stainless steel composites with varying TiN additions via spark plasma sintering (SPS). SPS process parameters such as sintering temperature, holding time, and heating rate were varied in this research using 2205-5TiN with the aim to obtain the best combination of parameters in terms of densification and hardness.

2. Materials and methods

2205 DSS (average particle size 22 μm, 96% pure, supplied by Sandvik Osprey Ltd, UK) and TiN (average particle size of 20 nm, 97% purity, supplied by Nanostructured & Amorphous Materials, Inc., USA) powders were utilized as the starting materials for this research. The chemical compositions of the powders are presented in Table 1.

The powders were mixed in a dry environment using the Turbula Shaker Mixer T2F at a mixing speed of 72 rpm for 8 h to ensure homogeneous mixing of powders. The morphology and phases of the mixed powders were examined with a field emission scanning electron microscopy (FESEM, JSM-7600F, JEOL, Japan) and X-ray diffraction (XRD) respectively.

The appropriate amount of mixed powders required in producing 20 mm diameter and 5 mm thick 2205-5%TiN nano structured composites were poured into a graphite die and then sintered using an automated spark plasma sintering machine (model HHPD-25, FCT GmbH, Germany). To ensure easy removal of sintered products and reduction of temperature in-homogeneities, graphite sheets were used to shield the powders from the die, upper and lower punches [25]. The mixed powders were sintered at different temperatures, times and heating rate as presented in Table 2. The composites were sintered in vacuum and sintering pressure was maintained at 50 MPa throughout the whole process [25].

Table 2 – Sintering process conditions (temp., time and heating rate) for development of SAF 2205-5TiN.

Sample designation	Sintering temp (°C)	Holding time (min)	Heating rate (°C/min)	Exp. density (g/cm^3)	% porosity
A	1000	10	100	7.31	4.07
B	1100	10	100	7.49	1.72
C	1150	10	100	7.51	1.44
D	1200	10	100	7.47	1.94
E	1150	5	100	7.44	2.28
F	1150	15	100	7.53	1.16
G	1150	15	50	7.46	2.02
H	1150	15	150	7.48	1.75

Please cite this article in press as: Oke SR, et al. Optimization of process parameters for spark plasma sintering of nano structured SAF 2205 composite. J Mater Res Technol. 2017. http://dx.doi.org/10.1016/j.jmrt.2017.03.004