Journal of Nuclear Materials 487 (2017) 348–361

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Production, microstructure and mechanical properties of two different austenitic ODS steels

T. Gräning*, M. Rieth, J. Hoffmann, A. Möslang

Institute for Applied Materials-Applied Materials Physics, Karlsruhe Institute of Technology, Eggenstein-Leopoldshafen, PO Box 3640, 76021 Karlsruhe, Germany

HIGHLIGHTS

- Comparison of the microstructure of extruded and hot-rolled ODS.
- Two-step mechanical alloying with ZrO_2 milling balls.
- Determination of precipitate size distribution depending on chemical composition and annealing times.
- Determination of the influence of sieving of mechanical alloyed powder on the near net shape products.
- Tensile tests of two different materials.

ARTICLE INFO

Article history:
Received 28 September 2016
Received in revised form
2 February 2017
Accepted 12 February 2017
Available online 24 February 2017

Keywords:
Mechanical alloying
EBSD
Oxide particles
Mechanical characterization
ODS steels
Annealing

ABSTRACT

This article is to summarize and examine processing parameters of novel developed austenitic oxide dispersed strengthened (ODS) steels. Comparing hot-rolled and extruded conditions after the same degree of deformation after and before annealing, are just some examples to give insights into the complex processing of austenitic ODS steels. One of the major drawbacks of the material is the more sophisticated production process. Due to a ductile matrix material with an increased stickiness during milling, a two-step milling procedure with the use of ZrO_2 milling balls was applied to raise the production yield and to use the abrasion of the ZrO_2 as an additional element to facilitate the formation of nano-sized precipitates. To get a better understanding how the different powder particle sizes after milling affect final properties, sieving was applied and revealed a serious effect in terms of precipitate size, distribution and mechanical properties. Grain sizes in relation to the precipitate size, annealing time and processing parameters were determined and compared to the mechanical properties. Hardness and tensile test have pointed out, that the precipitate size and number are more important in respect to the ultimate tensile strength than the grain size and that in this study hot-rolled material exhibited the better properties. The investigation of the microstructure illustrated the stability of precipitates during annealing at 1100 °C for 40 h. These heat treatments also led to a consistent grain size, due to the pinning effect of the grain boundaries, caused by precipitates.

1. Introduction

One of the main goals in nuclear materials research history is to develop steels for energy conversion under extreme conditions for future fusion or enhanced fission reactors [1–3]. Achieving this goal requires a high thermal stability of the microstructure to ensure outstanding mechanical properties and creep and corrosion resistance at elevated temperatures. Therefore, over the past few decades mechanical milling was optimized to enable the homogeneous formation of oxide dispersed strengthened (ODS) nanoparticles in a ferritic steel matrix to prevent grain coarsening and swelling during operation of nuclear power plants [4–10]. In recent years, austenitic steels have become more prominent as a competitor for ferritic steels due to the ODS induced mitigation of swelling and a better corrosion resistance in general [11–15]. However, the production process of austenitic steels is more difficult in comparison to ferritic steels. Because of a higher ductility of austenite during milling, powder sticks to the wall of the milling

* Corresponding author.
E-mail address: tim.graening@kit.edu (T. Gräning).

http://dx.doi.org/10.1016/j.jnucmat.2017.02.034
0022-3115/

container and to the milling media, which decreases the production yield and alternates the chemical composition [16–18]. However, recent studies have shown superior high temperature fatigue and mechanical behavior compared to ferritic steels [15,19,20]. Further publications have stated that the addition of Zr has a beneficial impact on the precipitate size and leads to the formation of complex Y-Zr-Ti-O cluster. Zr can also substitute Ti in the formation of these clusters [21–23]. For this reason, in addition to a lower adhesion of the powder to ZrO_2 and a lower abrasion with usually used 100Cr6 balls, milling balls made of ZrO_2 were used [18].

With structural information available, this publication targets on how the production process, chemical composition, and annealing time influence the distribution and size formation of precipitates. Furthermore, the influence of fabrication parameters, such as milling duration and sieving, on the microstructure and mechanical properties of two different austenitic ODS steels was examined. This publication addresses the following questions and is structured in the very same order:

(1) How does milling influence the chemical composition and mechanical properties?
(2) Does the abrasion of milling balls made of ZrO_2 contribute to the formation of precipitates?
(3) How does sieving of mechanical alloyed powder affect the properties of near net shape products?
(4) How stable are the nanoparticles at high temperatures?
(5) What are the differences in the microstructure and mechanical properties of hot-rolled and extruded materials?

2. Experimental

2.1. Material

The ODS steels were fabricated by mechanical alloying in a two-step process. In this publication two different chemical compositions are investigated, for simplicity labeled as A and B. The actual chemical compositions of the used powders are summarized in Table 1. In a first step, a ferritic master alloy was blended and ground with Fe_3Y powder for 30 or 40 h for material A or B, respectively. Fe_3Y was used instead of Y_2O_3 to decrease the oxygen content. 100Cr6 milling balls were used for the first milling step of both alloys to minimize the contaminations, due to abrasion. This step is followed by a second milling process with the addition of elemental nickel and a slight amount of elemental chromium powder. Chromium was added to increase the chromium content to the desired level. The second milling step was carried out with the utilization of ZrO_2 milling balls.

For all conducted milling processes of material A, a Simoloyer CM 01 mill by ZOZ with a load of 200 g per batch has been used. The first 40 h of milling of material B were conducted with a bigger ZOZ Simoloyer CM20 with a batch load of 3 kg prior to the process described above to be able to compare the contamination introduced by different sizes of milling vessels. Mixing and milling of all powders were handled in an argon atmosphere to prevent oxidation. The milling chamber was cleaned after each milling process.

The first milling step is utilized to dissolve the added Fe_3Y powder into the ferritic matrix. A production yield of almost 100 percent was achieved. The second step is performed with the purpose of alloying the elemental nickel and chromium powder with the obtained ferritic ODS steel powder. ZrO_2 milling balls were used to mitigate the adhesion of the ductile austenitic and nickel powder to the grinding media. Previous in-house research has shown, that the production yield of a two-step production compared to a one-step production, starting with an austenitic prealloy, is increased from 20 to 84,6% in average for 40 h of milling. This corresponds to an actual rise of 423%. A reduction of the milling time to 20 h in the one-step process increased the production yield to around 48%. But other researchers determined that 20 h of milling with the very same milling conditions as in this study are evidently not enough to distribute Yttrium homogeneously [24,25]. Table 2 shows the actual chemical composition after milling durations of a total of 60 and 70 h of alloy A and B, respectively.

After milling, powder B was sieved into 4 fractions and chemically analyzed along with non-sieved samples of powder A. Sieving was performed inside a glove box under the argon atmosphere with the help of a sieve stack. The chemical compositions were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES) followed by carrier gas hot extraction with an elemental analyzer. After sieving of material B in four size fractions, powders were filled in cans, degassed at 500 °C for 3 h and either hot isostatic pressed (HIP) with subsequent hot rolling or directly extruded without hot isostatic pressing. The HIP process took around five hours. It was conducted at 1150 °C for two hours with a pressure of 100 MPa. The other three hours are necessary to heat or cool the sample with a ramp of around 15 °C. After that, the material was hot-rolled. A total decrease in thickness from 36 mm to 8 mm in 5 rolling steps or 80 to 16 mm in diameter in direct extrusion were achieved, respectively. This is adequate to a deformation degree of −1.5 and −1.6. Hot rolling was performed in 5 passes with an intermediate heating of 10 min at the initial temperature of 1100 °C after each pass. Direct extrusions were completed in one-step with a processing temperature of 1100 °C.

Table 1
Actual chemical composition of used powders in wt.-% before milling.

Powder	Fe	Cr	Ni	Ti	Y	W	V	Mn	Zr	O	C	Comments	Particle size
Fe_3Y	Bal.	–	–	–	32.6	–	–	–	–	0.27	0.025	Added before 1st step	400 μm
Cr	–	Bal.	–	–	–	–	–	–	–	0.93	0.156	Added before 2nd step	250 μm
Ni	–	–	Bal.	–	–	–	–	–	–	0.19	0.008	Added before 2nd step	5 μm
Alloy A	Bal.	13.4	0.01	0.2	–	1.1	–	–	–	0.038	0.008	Base alloy for A	140 μm
Alloy B	Bal.	15.7	0.01	0.2	–	1.9	0.7	1.0	–	0.07	0.009	Base alloy for B	140 μm

Table 2
Actual chemical composition of powder A and B after milling in wt.-%.

Sample ID	Fe	Cr	Ni	Ti	Y	W	V	Mn	Zr	O	C	$1^{st}/2^{nd}$ milling step	Production yield after $1^{st}/2^{nd}$step
A (MSVI[a])	Bal.	16.7	13.7	0.20	0.19	0.9	–	–	0.45	0.38	0.08	30 h/30 h	100%/65–75%
B (MSVIII[a])	Bal.	16.1	14.0	0.15	0.19	1.6	0.7	0.7	0.25	0.25	0.03	40 h/30 h	100%/85–90%

[a] MS stands for milling study, mentioned for comparison reasons with Straßberger et al. [26].