Composition, microstructure and mechanical homogeneity evaluation of the Y-bearing 9Cr F/M steel fabricated by VIM & casting technique

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Abstract

Casting is suggested to be a promising method to produce the low-cost ODS steel with large volume and high throughput. However, the ingot homogeneity of the cast ODS steel was rarely reported. Recently, our group prepared a castable ODS steel, which exhibited long creep life of 3800 h under 650 °C and 120 MPa. Thus the purpose of this work is presenting the homogeneity of the castable ODS steel. Theoretical addition of Y was 0.1 wt% thereby the steel was named as 9Cr-10Y. Nine samples were machined from the top, middle, bottom, left, center, right regions of this plate then subjected to homogeneity analysis. Y content, prior austenite grain size and second phase size of the nine specimens were 0.023–0.034 wt%, 9.27–0.94 μm and 294–314 nm, respectively. Low coefficient of variation (Cv) of 14.5% for Y content, 2.3% for prior austenite grain size and second phase size indicated that the homogeneous composition and microstructure were achieved in the 9Cr-10Y plate. Besides, hardness fluctuated within a small range and all the Cv values were in the range of 0.8%–4.3%, which demonstrated that the hardness distribution along RD, TD, and ND was homogeneous. Furthermore, the 9Cr-10Y plate exhibited high strength of 765 MPa and high elongation of 18.9% as well as low DBTT of −40 °C.

1. Introduction

Oxide dispersion-strengthened (ODS) ferritic/martensitic (F/M) steels with 8–12 wt% Cr are considered as candidate structural materials for fuel cladding tubes in advanced fission reactors due to their excellent high-temperature mechanical properties and irradiation resistance [1, 2]. These superior properties are mainly attributed to a high number density of nanoscale oxide particles such as yttrium oxides (10^{23}/m^3) [3, 4]. In general, the typical manufacturing procedure of ODS steels is the powder metallurgy (PM), which possesses some drawbacks including complex, expensive and limited for industrial scale et al [5, 6]. Therefore, some alternative fabrication routes for ODS steel are being explored [7].

Vacuum induction melting (VIM) & casting is suggested to be a promising method to produce the low-cost ODS steel with large volume and high throughput. Recently, some ODS steels were fabricated by this technique and microstructure and mechanical properties were characterized in detail. Direct casting method [8] and intermediate alloy casting method [9] were developed by Han et al in the former method, the micro sized Y₂O₃ particles were directly added into the liquid T91 steel under electro-magnetic stirring. The results indicated that Y-bearing oxide particles with the size of 2–5 μm were obtained and the strength of the T91-ODS steel was increased by the Orowan strengthening mechanism. In the latter method, an intermediate alloy was firstly prepared by PM using pure Fe and Y₂O₃ powders and then added into the induction furnace and melted together with T91 steel. The results indicated that some nano Y₂O₃ particles were observed in the T91 matrix and the strength of the T91-ODS steel was increased by 9.6%. Besides, the oxygen carrier concept was also adopted to
prepared ODS steel by Nili-Ahmadabadi et al [10]. Firstly, Fe-10TiO2 (wt%) oxygen carrier discs were prepared by PM technique. Meanwhile, FeNiMn master alloy and Fe-20Y (wt%) interalloy was prepared in a vacuum arc melting furnace. Secondly, the master alloy and Y-rich interalloy were remelted to produce the FeNiMn-Y alloy. Finally, the FeNiMn-Y ingot was remelted together with Fe-10TiO2 to prepare the FeNiMn-Y-TiO2 alloy. The results indicated that two kinds of yttrium oxides including 626 nm Y2O3 and 11 nm Y2TiO5 were obtained in the matrix. Moreover, the addition of TiO2 as an oxygen carrier succeeded in the prevention of agglomeration and coarsening of yttrium oxides during the casting process. Briefly, the feasibility of VIM & casting method for ODS steel fabrication was identified by now.

Homogeneity is another important impact determining the application of ODS fabrication technique. However, the ingot homogeneity of the cast ODS steel was rarely reported. In the rare earth homogeneity experiments prepared ODS steel by Nili-Ahmadabadi et al [11–14]. For example, RE was prior to be accumulated at the bottom, edge regions in [15] and bottom-cone, middle-edge, riser regions in [16]. Similarly, bottom-cone segregation of Ce-bearing inclusions was also reported in 750 kg rare earth-treated steel by Waudby [17]. Recently, the rapid solidification production (RSP) technique was adopted to produce T91-Y2O3-YM ingots using colloidal Y2O3 dispersion and metallic yttrium (YM) [18]. Glow discharged optical emission (GDOE) spectroscopy was used to determine the homogeneity of yttrium oxides. Y signal was measured at the bottom, middle and top cross sections of the ingot. For the T91-Y2O3 steel, colloidal Y2O3 was only added in the T91 melt. The Y-bearing particles were distributed in-homogeneously throughout the matrix after solidification and the yttrium intensity was in the range of 0–0.06 wt%. In the case of the T91-Y2O3-YM, both colloidal Y2O3 and YM were added in the T91 melt. The distribution of Y-bearing particles was homogeneous and the Y signal exhibited high intensity of 0.22–0.3 wt%. The huge difference of the Y-bearing particle’s distribution between T91-Y2O3 and T91-Y2O3-YM YM was resulted from the addition of YM, which acted as a surface active can decrease the contact angle between Y2O3 and liquid steel.

With the development of VIM & casting technique, oxygen content and temperature of the molten steel can be controlled more accurately by now, which improves the feasibility of fabricating steel with the homogeneous distribution of RE and RE-bearing particles. Moreover, a kind of ODS F/M steel was prepared by Yan et al [19] and a large number of Y-Ti-O phases with the size of <5 nm were characterized by the scanning transmission electron microscope (STEM) and three dimensional atomic probe tomography (3D-APT) techniques. Furthermore, compared to the Y-free steel, the creep life of the ODS steel under 650 °C and 120 MPa was increased from 1800 h to 3800 h. Thus the purpose of this work is presenting the homogeneity of the ODS steel fabricated by VIM & casting method. Chemical composition, microstructure characteristics and mechanical properties of the top, middle and bottom regions in the plate will be characterized in detail. And the novelty of the work is presenting the distribution of Y in this castable ODS steel and evaluating the effect of Y distribution on the homogeneity of microstructure and mechanical properties.

2. Experiments

The 100 kg Y-bearing 9Cr F/M ingot was prepared by VIM & casting method and the main fabricating steps were listed as following: (1) 100 g Y powders (>99.9%) were placed at the bottom of the casting mold homogeneously (2) 100 kg Fe powders (>99.9%) and alloying powders (>99.9%) were induction melted under electro–magnetic stirring. (3) Pouring the liquid steel into the casting mold and the ingot was obtained after solidification. Subsequently, the ingot was subjected to forging, rolling and heat treatment. The heat treatment schedule was austenitizing at 1050 °C for 30 min followed by oil quenching then tempering at 750 °C for 2 h followed by air cooling. Nominal chemical compositions of the ingot are shown in table 1. The theoretical content of Y is 0.1 wt%, thus this steel was named as 9Cr-10Y. In order to evaluate the homogeneity along rolling direction (RD), transverse direction (TD) and normal direction (ND), nine samples including T1, T2, T3, M1, M2, M3, B1, B2 and B3 were cut from the 9Cr-10Y plate as shown in figure 1. T1, T2 and T3 samples were cut from the top region, M1, M2 and M3 samples were cut from the middle region, B1, B2 and B3 samples were cut from the bottom region. 1, 2, 3 referred to left, center and right of the plate, respectively.

Y content was measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES, Plasma1000). Each sample was measured four times at least. Microstructure was observed using laser scanning confocal microscope (LSCM, Olympus LEXTOLS40003D) and scanning electron microscope (SEM, LEO1450) equipped with energy dispersive spectroscopy (EDS). Before microstructure characterization, samples were mechanically polished and then etched by a solution consisting of 5 vol% HF and 95 vol% HNO3. Grain size and particle size were determined from LSCM and SEM images using Nano Measure software. Hardness was measured with a load of 187.5 kgf for 12 s using XHB-3000 Brinell hardiness testing instrument. Three measurements at least were adopted to ensure the hardness accuracy. Tensile tests were conducted at room temperature.
Table 1. Nominal composition of the 9Cr-10Y F/M steel, wt%.

| C  | Cr  | Mo | W  | Mn | Si | Ti | Ta | V  | N  | Y  | Fe |
|----|-----|----|----|----|----|----|----|----|----|----|----|
| 0.08–0.11 | 8.9–9.5 | 0.3–0.5 | 1.1–1.3 | 0.45–0.55 | 0.10–0.20 | 0.01–0.03 | 0.15–0.22 | 0.20–0.30 | 0.04–0.07 | 0.10 | Bal. |
temperature (RT) under a nominal strain rate of about $10^{-3}$ s$^{-1}$ using CMT6104 machine. The tensile specimens of dimensions 5 mm diameter and 25 mm length were machined from the sheet parallel to RD. Charpy V-notch impact tests were performed on the $10 \times 10 \times 55$ mm$^3$ specimens at RT, 0 °C, −20 °C, −40 °C, −60 °C, −80 °C and −100 °C, respectively using JB-30B machine.

Coefficient of variation ($C_V$) defined as the ratio between standard deviation ($SD$) and mean value ($M$) was used to evaluate the composition homogeneity, microstructure homogeneity and hardness homogeneity throughout the 9Cr-10Y plate. $C_V$ can be expressed by the equation (1) as following:

$$C_V = \frac{SD}{M} \times 100\%$$

The $SD$ of the Y content, prior austenite grain size, second phase size and hardness was evaluated using the following equation (2):

$$SD = \sqrt{\frac{\sum x^2}{n} - \left(\frac{\sum x}{n}\right)^2}$$

where $x$ is the variable measured and $n$ the number of measurements.

3. Results and discussion

3.1. Composition homogeneity

Measured Y contents of the nine specimens ranging from 0.023 to 0.034 wt% in the 9Cr-10Y plate are listed in table 2. Y content was the minimum on the T1 sample and the maximum on the M2 and B1 samples. The average Y contents at top, middle and bottom was 0.024, 0.031 and 0.030 wt%, respectively. Compared to the middle and bottom regions, the top one showed slightly lower Y content, which can be caused by the fact that Y powders were placed at the bottom of the mould before casting. The average Y contents at left, center and right was 0.029, 0.031 and 0.025 wt%, respectively. Although the Y content showed a trend of slightly increasing first and then decreasing from top to bottom and from left to right, $C_V$ of 14.5% for the nine Y contents indicated that Y content was homogeneous throughout the 9Cr-10Y plate. Usually, the data can be considered as unscattered when the $C_V$ is lower than 15%. Besides, it should be noted that $C_V$ of the top, middle and bottom samples, 7.9% for the left,
center and right samples and 14.5% for all the samples, it can be concluded that the homogeneity of Y content throughout the 9Cr-10Y plate was acceptable.

3.2. Microstructure homogeneity

Figure 2 shows the LSCM images taken from the 9Cr-10Y sheet at nine positions. As can be seen that tempered martensite was obtained after quenching and tempering. Prior austenite grain sizes, measured basing on these images, are listed in table 3. The average prior austenite grain size in the top, middle and bottom regions were 9.80, 9.55 and 9.57 μm, respectively. And the average prior austenite grain size in the left, center and right regions were 9.56, 9.51 and 9.84 μm, respectively. Obviously, the prior austenite grain sizes in different positions are approximately equal, which was well consistent with the low Cv values ranging from 0.3% to 3.0%.

Table 3. Prior austenite grain size of the top, middle and bottom samples in the 9Cr-10Y plate, μm.

| Samples | Top   | Middle | Bottom  | Mean  | Cv   |
|---------|-------|--------|---------|-------|------|
| Right   | 9.80  | 9.94   | 9.79    | 9.84  | 0.7% |
| Center  | 9.76  | 9.27   | 9.50    | 9.51  | 2.1% |
| Left    | 9.84  | 9.43   | 9.42    | 9.56  | 2.0% |
| Mean    | 9.80  | 9.55   | 9.57    | 9.64  | 1.2% |
| Cv      | 0.3%  | 3.0%   | 1.7%    | 1.5%  | 2.3% |

Figure 2. LSCM images of the top (T1-T3), middle (M1-M3) and bottom (B1-B3) specimens in the 9Cr-10Y plate.
Prior austenite grain size can be determined by the following factors: (I) Heating temperature and holding time. (II) Heating rate. (III) Compositions. Certainly, Y addition can facilitate grain nucleation and inhibit grain boundary movement, resulting in the refinement of the prior austenite grains. For the specimens with close chemical composition subjected to the same austenitizing temperature and holding time as well as the same heating rate, prior austenite grain size at the nine different positions was consistent naturally.

Figure 3 shows the SEM images of the top (T1-T3), middle (M1-M3) and bottom (B1-B3) specimens in the 9Cr-10Y plate. The white second phases were distributed in grain interiors and along grain boundaries. And there were no large inclusions due to the inclusion modification induced by RE [20]. The size of the second phase was measured basing on these SEM images and the results are shown in table 4. The average second phase size in the top, middle and bottom regions were 312, 306 and 303 nm, respectively. And the average second phase size in the left, center and right are 306, 306 and 309 nm, respectively. Obviously, there were no obvious differences in the particle size for these nine positions, indicating the uniform distribution of second phase along RD and TD. Figure 4 shows the three typical second phases in the matrix and the corresponding EDS results. For the 1# particle, intensive Y and O were also detected except for the matrix elements of Fe, Cr, which indicated that this particle can be yttrium oxide. For the 2# particle, Fe, Cr and C were detected simultaneously and Cr content was higher than the matrix, which indicated that this particle can be M23C6. For the 3# particle, Fe, Cr, C, Ta and V were detected simultaneously while Ta exhibited higher peak compared to Cr, which indicated that this particle can be MX. According to the EDS analysis, most of the particles were carbides and the rest were yttrium oxides. Besides, there should be a large number of nano Y-bearing particles in the high magnification TEM images according to the [19] but can’t be presented in this low magnification SEM images. Summarily, prior austenite grain size and
Table 4. Second phase size of the top, middle and bottom samples in the 9Cr-10Y plate, nm.

| Samples | Top    | Middle | Bottom | Mean  | Cv   |
|---------|--------|--------|--------|-------|------|
| Right   | 314    | 313    | 301    | 309   | 1.9% |
| Center  | 311    | 294    | 312    | 306   | 2.7% |
| Left    | 311    | 310    | 297    | 306   | 2.1% |
| Mean    | 312    | 306    | 303    | 307   | 1.2% |
| Cv      | 0.5%   | 2.7%   | 2.1%   | 0.5%  | 2.3% |

Figure 4. Typical SEM image of the B3 specimen (a). EDS result of the yttrium oxide particle (b). EDS result of the M$_2$C$_6$ particle (c). EDS result of the MX particle (d).

second phase size of the nine specimens demonstrated that almost homogeneous microstructure was achieved throughout the 9Cr-10Y plate.

3.3. Hardness homogeneity

3.3.1. Hardness distribution along ND

As mentioned above, nine samples were selected to characterize the composition and microstructure homogeneity of the 9Cr-10Y plate. In order to evaluate the mechanical homogeneity along ND, hardness was measured at the quarter-thickness, mid-thickness and three quarter-thickness positions from surface for each sample and the corresponding values are summarized in table 5. Obviously, hardness fluctuated within a small range and all the Cv values were in the range of 0.8%–4.3%, which indicated that the hardness distribution along ND in the 9Cr-10Y sheet was homogeneous.

3.3.2. Hardness distribution along RD and TD

The comparison of average hardness among the nine samples was used to evaluate the hardness homogeneity along RD and TD in the 9Cr-10Y steel as shown in table 6. The average hardness in the top, middle and bottom regions were 358, 360 and 350 HBW, respectively. And the average hardness in the left, center and right regions were 360, 353 and 355 HBW, respectively. Obviously, close hardness values were achieved along RD and TD, which was well accordance with the homogeneous distribution characteristics of Y content, prior austenite grain size and second phase size basing on the Hall-Petch relationship.
3.4. Mechanical properties

Based on the excellent homogeneity of Y content, prior austenite grain size, second phase size and hardness throughout the 9Cr-10Y plate, tensile properties were performed at three typical positions including T2, M2 and B2. Table 7 shows the tensile properties of the three samples at RT. The mean values of ultimate tensile strength (UTS), yield strength (YS) and total elongation (TE) were 765 MPa, 647 MPa and 18.9%, respectively. It can be seen that the three samples exhibited high strength and excellent plasticity simultaneously. The corresponding Cv of the UTS, YS and TE were 2.2%, 2.4% and 4.4%, respectively. These low Cv values also indicated that the tensile properties at the top, middle and bottom regions were homogeneous. In addition, UST, YS and TE were 627 MPa, 483 MPa, 21.0% for the 9Cr-2WVTa-0.15Y steel [21] and 675 MPa, 538 MPa, 23.5% for the CLAM-0.22Si steel [22]. Obviously, compared to the 9Cr-2WVTa-0.15Y and CLAM-0.22Si steels, the 9Cr-10Y steel exhibited higher strength and close plasticity.

Due to the limitation of sample quantity, Charpy impact tests were only conducted on the M2 sample at temperatures ranging from −100 °C to RT. Figure 5 shows the absorbed energy as the function of testing temperature and the Charpy impact curve was obtained basing on the Boltzmann fitting formula. The upper shelf energy (USE) was about 272 J and the ductile-to-brittle transition temperature (DBTT) was about −40 °C. DBTT was −60 °C for the Eurofer 97 steel [23] and −30 °C for the CLAM-0.22Si steels [22]. Compared to the Eurofer 97 and CLAM-0.22Si steels, the 9Cr-10Y steel exhibited moderate DBTT.

4. Conclusions

A 9Cr F/M steel with the theoretical Y addition of 0.1 wt% was prepared by vacuum induction melting, casting, forging, rolling and heat treatment. Subsequently, nine samples were machined from the top, middle, bottom regions and left, center, right regions of this 9Cr-10Y plate. Finally, Y content, prior austenite grain size, second phase size and hardness of each sample were characterized in detail to evaluate the composition homogeneity, microstructure homogeneity and mechanical homogeneity of the 9Cr-10Y plate. The main conclusions derived from the experimental work are as follows:

### Table 5. Hardness distribution of the nine specimens along ND in the 9Cr-10Y plate, HBW.

| Samples | Value (HBW) | C_v |
|---------|-------------|-----|
| T1_1/4  | 364         | 2.1%|
| T1_1/2  | 346         | 1.2%|
| T1_3/4  | 356         | 1.7%|
| M1_1/4  | 366         | 0.8%|
| M1_1/2  | 373         | 1.2%|
| M1_3/4  | 369         | 3.3%|
| B1_1/4  | 338         | 3.5%|
| B1_1/2  | 362         | 4.3%|
| B1_3/4  | 366         | 3.5%|

| Samples | Value (HBW) | C_v |
|---------|-------------|-----|
| T2_1/4  | 342         | 1.2%|
| T2_1/2  | 350         | 1.7%|
| T2_3/4  | 351         | 1.2%|
| M2_1/4  | 338         | 3.3%|
| M2_1/2  | 355         | 1.2%|
| M2_3/4  | 366         | 3.3%|
| B2_1/4  | 338         | 4.3%|
| B2_1/2  | 376         | 4.3%|
| B2_3/4  | 359         | 4.3%|

### Table 6. Hardness distribution of the nine specimens along RD and TD in the 9Cr-10Y plate, HBW.

| Samples | Top | Middle | Bottom | Mean | C_v |
|---------|-----|--------|--------|------|-----|
| Right   | 372 | 358    | 336    | 355  | 2.8%|
| Center  | 348 | 353    | 358    | 353  | 1.2%|
| Left    | 355 | 369    | 355    | 360  | 1.8%|
| Mean    | 358 | 360    | 350    | 356  | 1.3%|

### Table 7. Tensile properties of the T2, M2 and B2 samples at RT.

| Samples | Value (MPa) | C_v |
|---------|-------------|-----|
| UTS     | 765          | 2.2%|
| YS      | 647          | 2.4%|
| TE      | 18.9         | 4.4%|

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(1) Y contents of the nine specimens were in the range of 0.023–0.034 wt% and the Cv of 14.5% demonstrated that Y content was homogeneous throughout the 9Cr-10Y plate.

(2) Prior austenite grain size and second phase size of the nine specimens were 9.27–0.94 μm and 294–314 nm. Low Cv of 2.3% for prior austenite grain size and second phase size indicated that homogeneous microstructure was achieved in the 9Cr-10Y plate.

(3) Hardness fluctuated within a small range and all the Cv values were in the range of 0.8%–4.3%, which indicated that the hardness distribution along RD, TD, and ND in the 9Cr-10Y sheet was homogeneous.

(4) The 9Cr-10Y plate exhibited high strength of 765 MPa and high elongation of 18.9% as well as low DBTT of −40 °C.

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Figure 5. Charpy impact curve of the M2 sample machined from the 9Cr-10Y plate.
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