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Evaluation of microstructure high chrome austenitic stainless-steel grade 253MA after creep test at temperature of 700°C

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Abstract: High Chrome Austenitic Stainless-Steel grade 253 MA is a material that widely used for high temperature. This is due the fact this material has excellent mechanical properties and creep resistance. However, changes in microstructure can occur in long-term use, which will affect the creep resistance (shortened service life of the material). The microstructure of High Chrome Austenitic Stainless-Steel 253 MA creep test specimens was investigated. Creep testing at a temperature of 700 °C with a loading of 150 MPa was carried out. The cold rolling process with 53% reduction in thickness was applied followed by annealing at 900°C, 1000°C, and 1100°C for 3600s to obtain different grain size. Grain size after annealing and after creep test was measured to see the effect of annealing temperature on the grain size of tested steel and to see its effect on creep resistance based on the creep test conducted. Grain size and morphology of the phase after creep test were observed by scanning electron microscope and optical microscope.

Keywords: Annealing; Austenitic stainless steel; Creep; Grain size; 253MA

1. Introduction

High Chrome Austenitic Stainless Steel 253MA is one of the austenitic steels that has excellent strength and oxidation resistance even at high temperatures (around 2000°F). Because this steel contains more nitrogen and less chromium, the alloy is less susceptible to sigma phase embrittlement than 25Cr/20Ni steel. Due to its good mechanical properties, this alloy is used for burner components, furnace tubes, furnace rollers and other items that operate at relatively high temperatures.

The alloy also contains a large amount of carbon and a small amount of a rare earth material (cerium) which provides excellent creep resistance properties. Rare earth materials are known to reduce sulfur and oxygen content which will affect the increase in creep resistance and heat workability (Cosandey et al., 1983). The creep resistance of this material is also due to its high carbon and nitrogen content. This has been studied in previous papers (Maode & Sandström, 1988).

In the use of high-temperature materials, diffusion-controlled microstructural transformation will occur. The most common type of reaction is the precipitation of an unwanted phase, which in addition to lowering the corrosion resistance, also causes a decrease in the toughness/ductility of the material. From the previous study, $M_{23}C_6$, -nitride, and -phase fine precipitate particles appeared after long-term creep testing at grain boundaries (Maode & Sandström, 1988) which could reduce the toughness of the material. Long-term use can also cause sedimentation. In addition to the phase, inclusions in the steel can also affect the characteristics of the steel. For example, increasing inclusions in the form of sulfur in 253MA austenitic steel material can reduce creep ductility (SandströmY., 1988). At elevated temperatures, brittle cracking can occur, whereby creep cavities and cracks form around these inclusions thereby contributing to a reduction in ductility. Sulfides at the grain boundaries in low alloy steels are the most frequently used nucleation sites which eventually lead to creep cavities (Cane & Middleton, 1981). These changes in microstructure can affect creep resistance and greatly affect the service life of the material.

Based on the key issues mentioned above, the aim of this study was to evaluate the microstructure of the long-term creep test. The morphology, distribution, and grain size after creep test under 150 MPa pressure at 700 °C under various initial conditions of different grain sizes resulting from annealing (different heat treatment temperatures) were also investigated.

2. Methods

The investigated steel was received in form of 60.33mm diameter pipe and 3.9mm thickness. The composition of this steel is shown in Table 1. The initial microstructure consists in equiaxed grains and annealing twins.

The steel was cut using an Electrical Discharge Machining (EDM) wire cutting machine (Taizhou Jiangzhou CNC Machine Tool company, Jiangsu, China) to form a cold-rolled sample with a length of 165 mm, a width of 25.7 mm, and a thickness of 3.9 mm. Cold rolling was carried out to reduce the thickness of the sample to approximately 53% so that the final dimensions achieved were a length of 267 mm, a width of 26 mm, and a thickness of 2.3 mm. Then, the steel was formed into creep rupture test specimen with a gauge section of 25 mm length.

The tested steels were then put into the tubular furnace (Nabertherm GmbH RSH 50, Lilienthal, Germany) for annealing process. The annealing process on the tested steel was carried out with a time duration of 3600 seconds with variations in annealing temperature of 900 °C, 1000 °C, and 1100 °C. After that, it is cooled by flowing hydrogen gas. The technical specifications of the cold rolling, tubular furnace, and annealing processes are explained in references (Anwar et al., 2021; Melinia et al., 2022).

After heat treatment, creep rupture tests were carried out at 700°C with stress 150 MPa using ZwickRoell test equipment (ZwickRoell GmbH & Co. KG, Ulm, Germany). The microstructures of the steel were observed before and after creep testing using optical microscopy (OM) and Scanning Electron Microscopy (SEM). OM was carried out on Olympus DSX-510 and SEM investigations were done on a JEOL Type JSM 6390 A test equipment (JEOL, Ltd, Tokyo, Japan). Before being observed through an optical microscope, the tested steel must be prepared first. The tested steels were sanded using grids of 200, 400, 600, 1000, and 2000. Then, the tested steels were polished using 5nm, 3.5nm, and 1nm diamond paste. Furthermore, the tested steel was etched using 20 mL of HNO_3 plus 60 mL of HCl with the ASTM E 407 etching method to be able to perform optical microscope observations with magnifications of 100x, 200x, 400x, 600x, and 1000x. Grain size measurements were performed on optical micrographs in accordance with Circular Intercept method (ASTM E112). ImageJ software was employed to measure grain size.

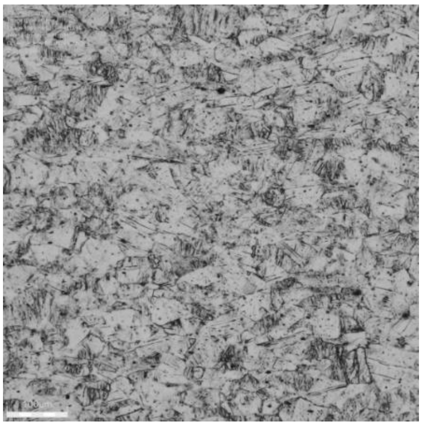
Table 1. Chemical composition (wt%) specification of the High Chrome Austenitic Stainless-Steel grade 253MA

C	Si	Mn	P	S	Cr	Ni	N	Ce
0.079	1.422	0.51	0.03	<0.005	22.06	10.86	0.384	0.03

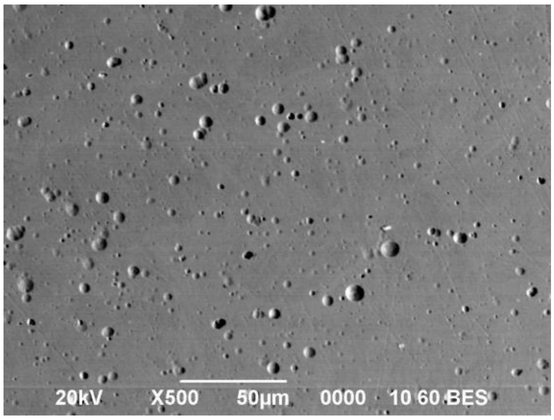
3. Results and discussion

Initial microstructures

Mechanical treatment carried out caused changes in the microstructure. The initial tested steel grain measurement result was 23.56 ± 1.1329 µm, after cold rolling with a deformation of 53% the grain size became 12.68 ± 1.1381 µm. After the cold rolling process, there is a change in grain shape from equiaxed to elongated grain. Figure 1a-b shows the microstructure of tested steel after cold rolling formed flatter and elongated grains in the rolling direction. Precipitation and twinning were visible on the microstructure of tested steel after cold rolling.



(a)



(b)

Figure 1. Showing the microstructure of high chrome 253MA steel after cold rolling (a) using optical microscope (b) using SEM

Austenite grain growth

The austenite grain growth of tested steel under annealing at temperature of 900, 1000, and 1100 °C was observed by using optical microscope. The grain size increased as annealing temperature increased and the austenite grain size seems to be quite large at an annealing temperature of 1100 °C compared with at the other temperature, as can be seen in Fig 2.

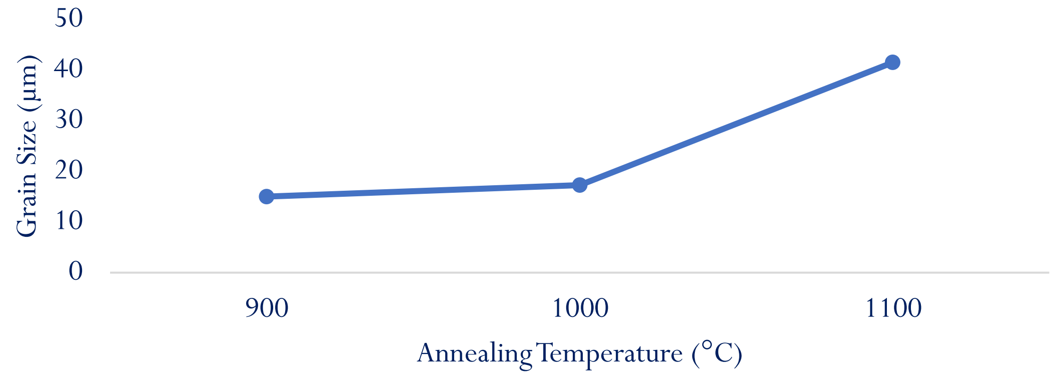


Figure 2. Austenite grains growth of high chrome 253MA steel with variations in annealing temperature

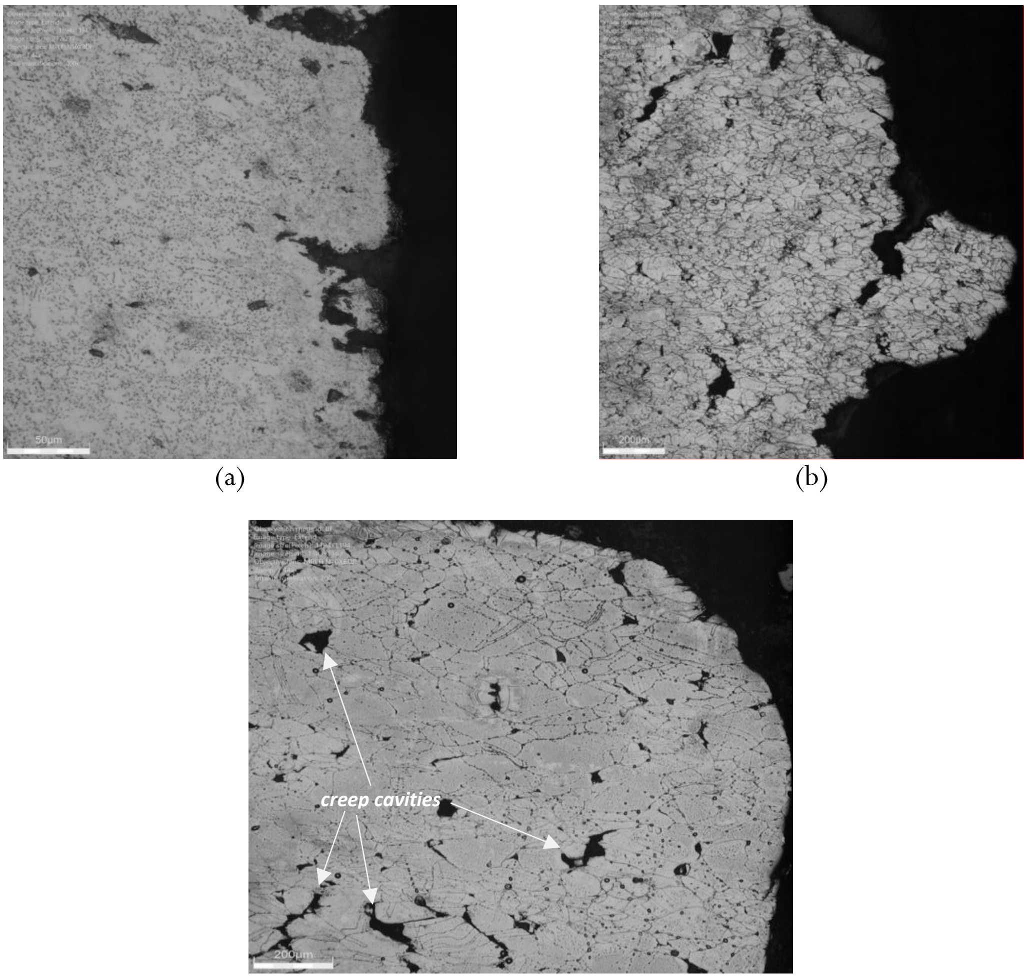
The result of precipitate of cold rolled tested steel and after annealing tested steel. The elements C, Cr, Ni, Ce, and Fe may form metal carbide ($M_{23}C_6$) precipitates in the austenite grains, may be seen in Table 2.

Table 2. Showing EDAX analysis of high chrome 253MA steel precipitate

Sample	(%wt)							
	C	O	Si	Cr	Mn	Fe	Ni	Ce
Cold Rolled	33.26	-	1.21	15.04	-	43.76	6.73	-
Anneal 1100 ℃	27.29	6.21	-	16.1	-	43.21	6.27	0.45

Creep rupture test microstructures

Figure 3 show the microstructures of tested steel after performing the creep rupture test under annealing temperatures of 900, 1000, and 1100 °C. The figure shows that after the creep rupture test, there were many changes in the shape of the grains from the previously very visible twinning of the grains to no visible twinning and the cavities appeared around grain boundary areas. The fracture that occurred in the tested observed seem to be as ductile fracture, as can be seen in Figure 4 below is look like dimples shape in the fracture surface and the size tend to be decrease as annealing temperature increase.



(a) (b) (c)

Figure 3. Showing the microstructure of high chrome 253MA steel after performing a creep test at annealing temperature of (a) 900 °C (b) 1000 °C (c) 1100 °C

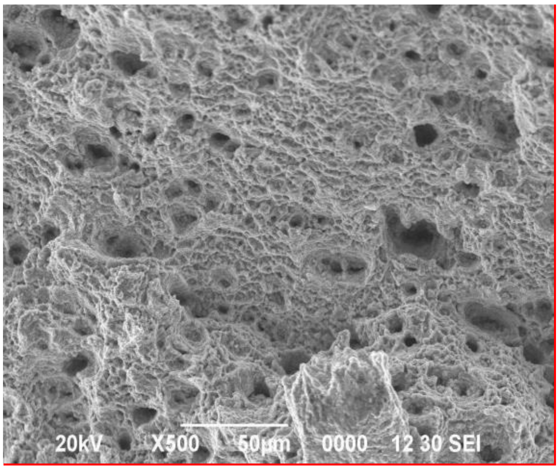


Figure 4. Showing the fracture surface of tested steel after creep test with annealing temperature of 1100° C using SEM

Creep rupture test behavior

Figure 5 shows the creep rupture behavior of tested steel under various annealing temperature. Tested steel with annealing 1100°C which has the coarse grain size has took a longer time fracture compared with the others and the time fracture around 282.07 hours was recorded.

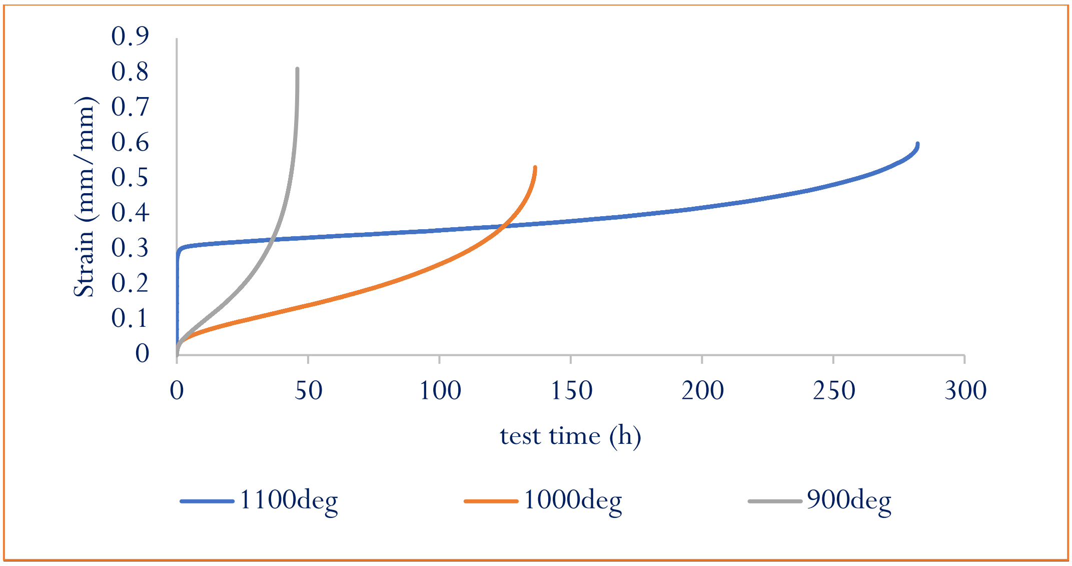


Figure 5. Effect of annealing time on the creep rupture behavior of high chrome 253MA steel

From the results of microstructural observations, it was shown that the mechanical and heat treatment carried out during experiment may be caused the changes in the microstructure. After cold rolled deformation, the grain size decreases and then increases as annealing time increased. In seem that under annealing temperature at below of 1100°C, the precipitates may not be completely dissolved and pin the grain boundaries so that the growth of austenite grains to be slow. While at a higher temperature, the precipitate has poor stability, so the pinning effect reduced (Ji et al., 2019). In tested steel at a annealing temperature of 1100°C, the percentage of carbon atoms decreased slightly. This indicates that the higher annealing temperature caused metal carbide precipitates to dissolve into the austenite grain.

The measurement results of the creep test tested steels with annealing temperatures of 900, 1000 and 1100 °C were 16.34 ± 0.7858 µm, 18.27 ± 1.8505 µm and 43.63 ± 1.3642 µm respectively. The difference in grain size in the tested steel with the annealing process at 1100 °C is quite large, as was the case for the grain size before the creep test was carried out. Coarsening of the grain size of the tested steel may occur due to the recovery of dislocations along the grain boundaries and the combination of the two sub-grain boundaries which may occur at the meeting point of the three grains. This recombination occurs due to dislocation movement and causes the loss of grain boundaries in the area (Nie et al., 2013). Meanwhile we can see in Figure 3a-c that the precipitate formed at the grain boundaries is getting bigger. Repeated dislocation movements at grain boundaries causing local melting also led to more and more precipitate deposition along grain boundaries. From the microstructure observation of tested steel after creep rupture test, we can see that there is precipitate in the cavity. The precipitate in the form of $M_{23}C_6$ carbide is one of the precipitates that has the potential to become a nucleation site (Nie et al., 2013). This is a location that can initiate failure. In tested steel with higher annealing temperatures, the cavities that appear are larger, this is because the longer the creep occurs, the larger the cavities that are formed.

Figure 5 shows the relation between grain size and creep rupture behavior. The primary creep stage at the annealing time of 900°C showed a higher elastic strain than the other annealing times. This is different from the previous study that both the rupture life and the elongation tend to increase as the grain size (Tanaka et al., 2000). The secondary creep stage at the coarser grain size was longer than other, which was caused by a high resistance to creep rupture. Normally it is accepted that the finer grain sizes lead to poor creep properties in material. It may because grain boundary area being more in fine grained material. Grain boundary sliding is one of the dominant mechanisms for creep to occur.

4. Conclusion

After annealing process, the grain size increases with the increasing of annealing temperature. Grain growth at annealing temperature below 1100°C showed a slight increase, due to the presence of precipitates which resulted in slow grain growth. At higher temperature of annealing, the pinning effect reduced. Coarsening of the grain size of the tested steel after creep test may occur due to the recovery of dislocations along the grain boundaries and the combination of the two sub-grain boundaries which may occur at the meeting point of the three grains. The coarser grained tested steel has a higher resistance to creep rupture than other, but the highest elastic strain was achieved in fine grained tested steel.

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