Influence of high temperature ageing on the toughness of advanced heat resistant materials

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Abstract Advanced biomass, biomass co-firing in coal-fired and future advanced USC coal-fired power plants with high efficiency require the materials to be used at even higher temperature under higher pressure. The reliability and integrity of the material used are therefore of concern. In this study, the influence of ageing at temperatures up to 700°C for up to 3 000 hours on the toughness of two advanced heat resistant austenitic steels and one nickel alloy are investigated. The influence on toughness due to differences in the chemical composition as well as the combined effect of precipitation and growth of the precipitates has been analysed by using SEM techniques. The fracture mechanisms that are active for the different ageing treatments are identified as a function of temperature and time. Local approach methods are used to discuss the influence of the precipitation and growth of precipitates on the toughness or fracture in the different aged materials.

Keywords High-temperature, ageing, toughness, austenitic stainless steel, nickel base alloy

1. Introduction

Renewable and more efficient energy resources such as biomass, biomass co-firing in coal-fired power plants and future advanced ultra-super critical (AUSC) coal-fired power plants for sustainable energy production are greatly desired. To increase efficiency higher temperature and pressure are used. Today austenitic stainless steels are commonly used as structural material in these power plants and they need to have good reliability and structure integrity with respect to the higher temperature and pressure [1, 2].

High-temperature long term ageing provides a range of precipitates that differ with ageing conditions and alloy composition. Precipitates from ageing of austenitic stainless steel and nickel base alloys have been studied by others [3-7]. Austenitic stainless steels are expected to provide reliable service for 30 years or more [3]. Their detailed mechanical properties are dependent on the stability of the microstructure particularly the formation, dissolution and coarsening of participates. In this study the influence of precipitation and growth of precipitates on toughness due to different compositions and high-temperature treatment are investigated.

2. Materials and experimental details

2.1. Materials

The experiments were conducted on two austenitic stainless steels (AISI 304 and Sanicro 28) and one nickel base alloy (Alloy 617), supplied by Sandvik Material Technology, see table 1 for nominal compositions in wt%. The materials are heat treated for normalizing.

Table 1. Compositions for the different materials in wt%.

Material	C	Si	Mn	Cr	Ni	W	Co	Cu	N	Mo	Fe
AISI 304	0.06	0.3	1.4	18.5	8.2	-	-	-	-	-	Bal.
Sanicro 28	0.02	0.43	1.83	27	31	0.02	0.09	0.9	0.05	3.4	Bal.
Alloy 617	0.1	-	-	22.5	53.8	-	12	-	0.5	9	1.1

2.2. Ageing and toughness tests

Standard sample with a dimension of 50x10x10mm (EN 10045-1) was used. The samples were aged at 650°C and 700°C in air-environment for 1000 and 3000 hours before the toughness testing. Impact toughness test has been performed at room temperature (RT), two to three samples at each temperature were tested.

2.3. Scanning electron microscope investigation

For the microstructural investigation of deformation and fracture behaviour scanning electron microscopy (SEM), electron channeling contrast imaging (ECCI) and an energy dispersive x-ray system (EDS) were used. An HITACHI SU-70 FEG-SEM and a Zeiss Sigma FEG-SEM were used to perform these microstructural investigations. ECCI uses the orientation dependence of the crystal lattice planes with respect to the incident electron beam to generate an image using backscattered electrons. Dislocations create small distortions in the crystal lattice which affect the backscattered electron intensity, allowing the imperfection to be imaged as a contrast change [8].

3. Results

3.1. Toughness test results

Table 2 shows the results of the impact tests, which reveals two different responses. The nickel base alloy, Alloy 617, is ductile at all ageing conditions and is more ductile than both austenitic stainless steels at all conditions. It seems that the impact toughness increases with both increasing temperature and longer times, but is affected more by temperature according to the energy differences in table 2. For the two austenitic stainless steels the impact toughness decreases with both increasing temperature and longer times. For AISI 304 the temperature has the biggest influence on the impact toughness. The material becomes brittle when the material has been aged at 700°C. Sanicro 28 has relatively high impact toughness at both 650°C and 700°C for 1000 hours, but the impact toughness decreases with increasing temperature, especially at 700°C, the material becomes brittle for about 3000 hours.

Table 2. Energy for each alloy at different ageing times and temperatures.

Material	Energy [J] 650°C & 1000h	Energy [J] 650°C & 3000h	Energy [J] 700°C & 1000h	Energy [J] 700°C & 3000h	Energy [J] Reference (RT)
AISI 304	75	31	10	6	243
Sanicro 28	100	64	50	5	>300
Alloy 617	127	137	137	151	>300

3.2. Precipitates and its influence on the toughness

The toughness is strongly influenced by the precipitates formed during the high-temperature ageing process. Table 3 shows the precipitates appearing in Sanicro 28 at the four ageing conditions. Comparing table 2 and 3 it is clear that the largest impact toughness decrease for Sanicro 28 (50 J to 5 J) is due to increasing amount of sigma phase, σ, with needle shape. The σ-phase is a brittle tetragonal phase and can differ relatively much in composition and occur at different ageing conditions depending on chemical composition of the aged material [3, 9]. This indicates that both temperature and time are important for the impact toughness of Sanicro 28. The Cr,Mo-rich carbides in Sanicro 28 is probably $M_{23}C_6$ and/or M_6C depending on ageing condition [3, 10]. Intergranular carbides can be observed in all samples of the tested materials and occur before intragranular precipitation. AISI 304 show similar precipitation progress as Sanicro 28, but the amount of σ-phase seems to be larger already from the first ageing condition and the needle shaped σ-phase appear at all ageing conditions except of 650°C and 1000 hours, AISI 304 also has a larger amount of σ-phase with needle shape at each ageing condition [3, 11, 12]. For the nickel base alloy Alloy 617, γ´ precipitate is a main precipitate which grows at higher temperature and longer times, which probably is the reason for the increase in impact toughness with both temperature and time. Compared to non-aged material ductility decreases when the material is aged due to precipitates that increases the strength. However, when the precipitates grow larger and are evenly distributed the strength decreases and the ductility increases again. Other precipitates that are common but not detected by the EDS investigation in this study due to their small size in these ageing conditions is carbides as $M_{23}C_6$ [6, 7, 13].

Table 3. Precipitates of Sanicro 28 for different ageing conditions.

Ageing condition	Precipitates	Location of precipitates	Shape/amount
650°C & 1000h	Cr,Mo-rich carbides σ	Intergranular Intergranular	Rectangular/large Round/small
650°C & 3000h	Cr,Mo-rich carbides σ	Intergranular Intergranular	Rectangular/large Round/small
700°C & 1000h	Cr,Mo-rich carbides σ	Inter- and intragranular Inter- and intragranular	Rectangular/large Round/small, needle/small
700°C & 3000h	Cr,Mo-rich carbides σ	Inter- and intragranular Inter- and intragranular	Rectangular/large Round/large, needle/large

Figure 1 illustrates how the amount of σ phase affect the impact toughness for these three materials. The amount of precipitate is given by ThermoCalc-simulations [14]. The impact toughness is decreasing when the fraction of σ phase increases. There are notable differences for the two ageing conditions. At the ageing condition, 700°C for 3000h, the impact toughness is much lower than other conditions. One of the reasons is that this figure only considers the effect of σ-phase since the other phases can also play important roles, another reason is the shape of the precipitates that changes from rectangular to needle shaped. Also the ThermoCalc-simulation is only valid for equilibrium which not necessarily the samples that have been aged and then impact tested are.

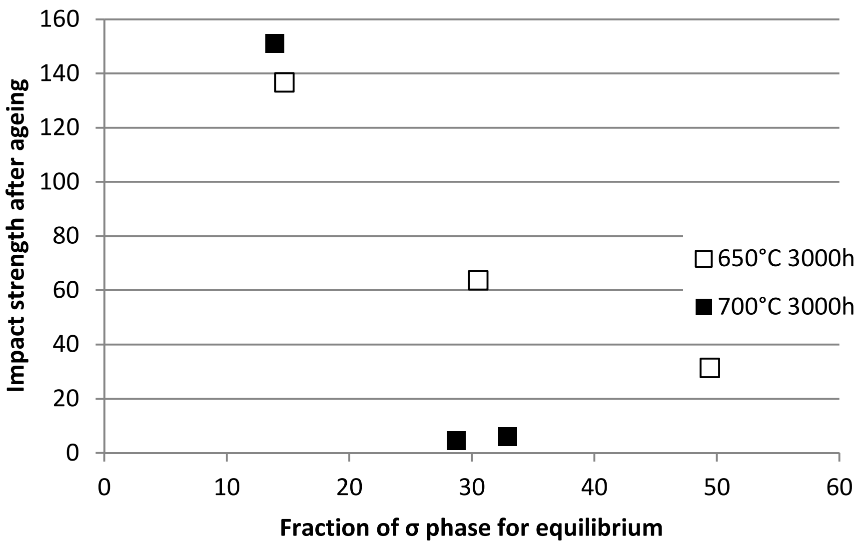

Figure 1. Show how the impact toughness is influences by the amount of σ phase at equilibrium after ageing at 650°C and 700°C for 3000 hours.

3.3. Fractures

The microstructure investigation reveals that the precipitates strongly affect the fracture behaviour. AISI 304 and Sanicro 28 show intergranular fracture after they have been aged at 650°C for 3000 hours (Fig. 2). However, the fracture morphology is different. Fracture surfaces in AISI 304 shows small and shollow dimples, while fracture surfaces in Sanicro 28 are rather smooth. Fracture surfaces in the nickel base alloy Alloy617 are still ductile and intergranular fracture is rarely seen.

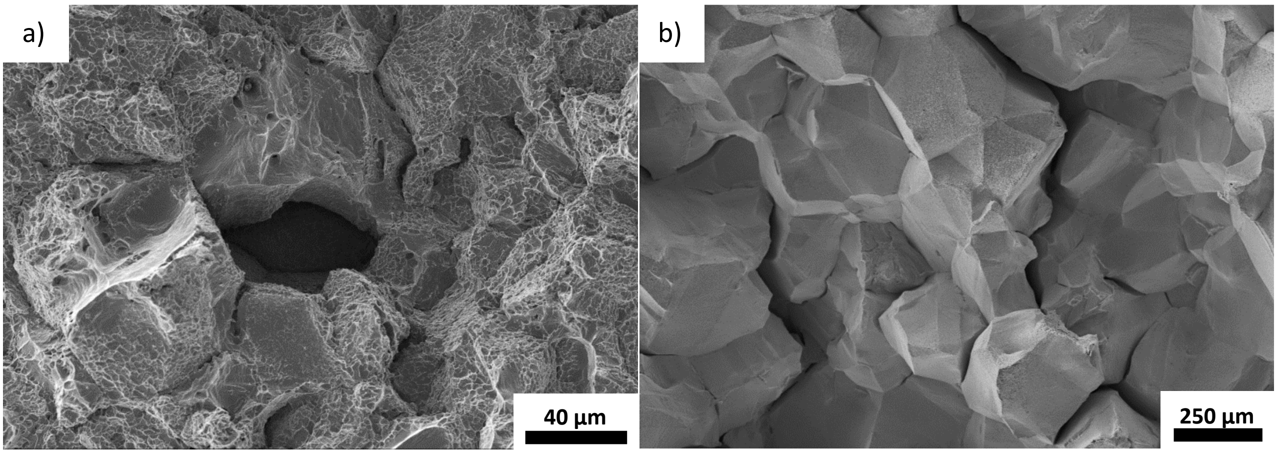

Figure 2. Pictures showing fracture surfaces from the impact tested samples, a) AISI 304 and b) Sanicro 28, both materials have been aged at 650°C for 3000h.

At 700°C, cleavage fracture can be observed in the stainless steels aged for 3000 hours (fig. 3). Typically step like or layer cleavage planes are found and some dimples can still be observed at the fracture surfaces.

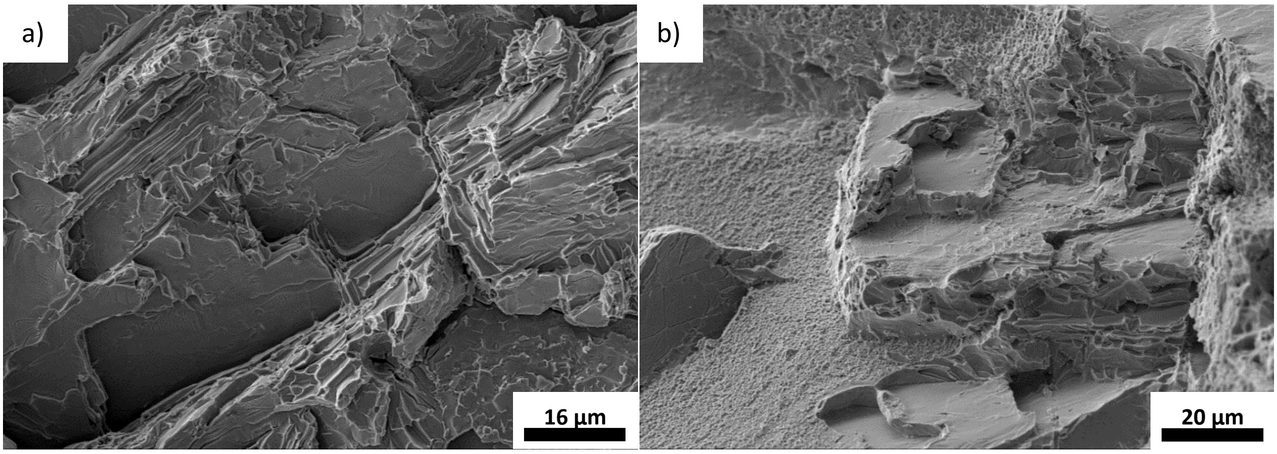

Figure 3. Cleavage fracture and elongated dimples from the impact tested samples after ageing at 700°C for 3000h, a) AISI 304 and b) Sanicro 28.

4. Discussion

As shown previously, ageing at 650°C and 700°C will lead to precipitation of different intermetallic phases with different morphologies. Since these materials are hard, stress concentration can form around the particles during creep or other deformation process, which leads the formation of local cracking or fracture [15]. Four types of cracks can be identified as shown in Fig. 4.

I. Cracks form at the particles within the grains. Due to stress concentration, this type of crack can induce cleavage fracture if the matrix is hard or brittle.
II. Cracks form at the particles at the grain boundaries. This may cause intergranular cracking if the matrix is tough or cleavage fracture if the matrix is brittle.
III. Cracks form along the needle shaped particle. This type of crack can create a high stress concentration, which can induce cleavage fracture if the matrix is not tough enough.
IV. Cracks form outside of grain boundary due to the shift of the cracking particles. This can cause both intergranular cracking and cleavage cracking.

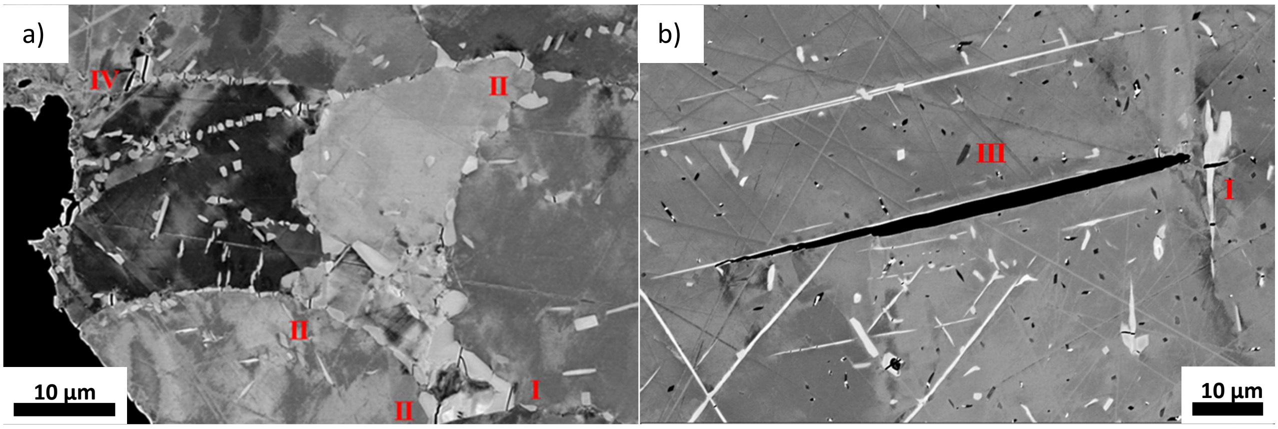

Figure 4. Precipitation induced cracks, a) AISI 304 aged at 650°C for 3000 hours and b) Sanicro 28 aged at 700°C for 3000 hours.

For the 304 material, needle shaped sigma phase can precipitate during ageing at 700°C for 3000h (Fig. 5a), which can form type III cracks during the impact toughness testing and lead to the formation of step type cleavage (Fig. 5b). This leads to low impact toughness.

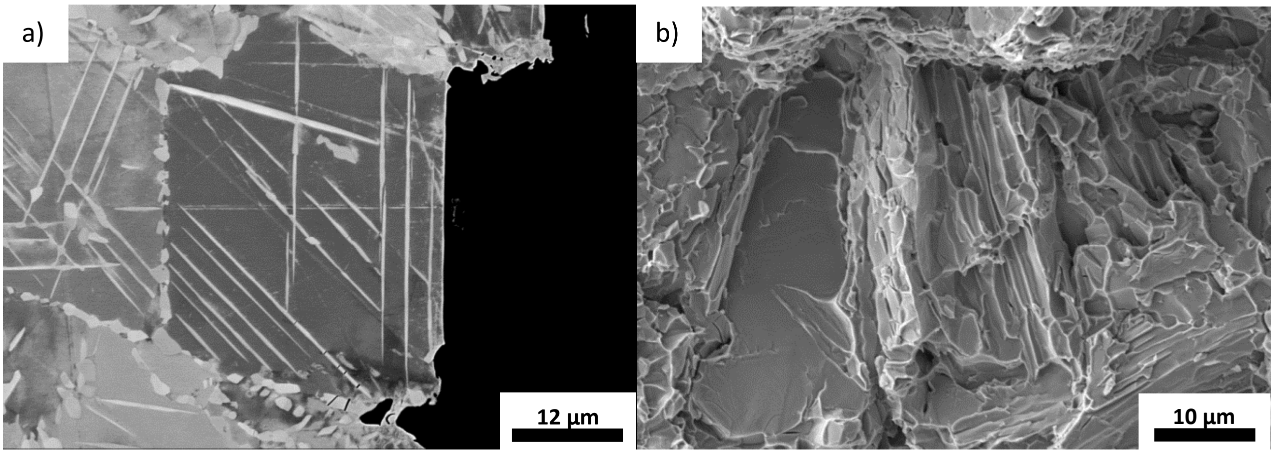

Figure 5. a) Precipitates in the aged 304 material at 700°C for 3000h and b) fracture after the impact toughness testing.

At 650°C, grain boundary carbides are the main precipitates (fig. 6a) and type II cracks can form during the impact toughness testing, which can lead to the formation of intergranular cracking (fig. 6b). Relatively low impact toughness can then be obtained.

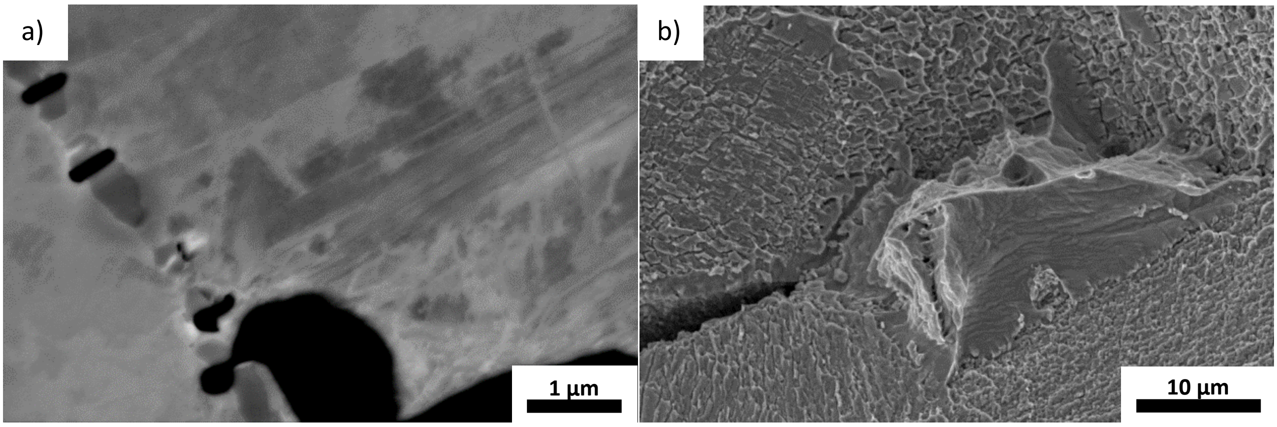

Figure 6. a) Precipitates in the aged Sanicro 28 material at 650°C for 3000h and b) fracture after the impact toughness testing.

Alloy 617 is a nickel based alloy and $Ni_3(Al,Ti)$ type of γ' is the main precipitate in the grains (Fig. 7a), but grain boundary carbides can also be observed [7]. In this case type II cracks can be observed and partial intergranular cracking can occur (Fig. 7b). Since the carbide density at the grain boundaries is relatively low, the material can still show good impact toughness.

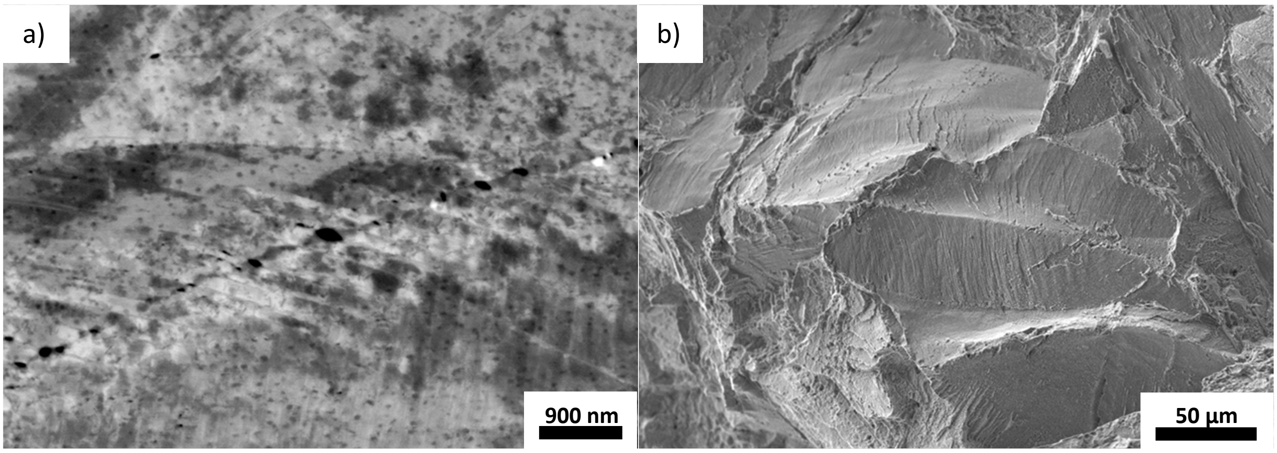

Figure 7. a) Precipitates in the aged Alloy 617 material at 700°C for 3000h and b) fracture after the impact toughness testing

The results and discussion above shows that initiation and propagation of fracture in the aged austenitic stainless steel is local. They can behave very differently in different materials, as explained by the four different crack-types. Local approach to fracture should be applied by considering the heterogeneous mechanical behaviors in these two phases.

5. Conclusions

The fracture initiation and propagation in the aged austenitic stainless steel is very local. They behave very differently in these materials at high temperature due to different chemical compositions affecting nucleation, growth and shape of precipitates. The brittle σ-phase can appear in the austenitic stainless steel after 1000 hours at 650°C and then increases in amount. The amount and shape have strong effect on the fracture behaviour, where needle shaped σ-phase which mostly appear at high temperature (700°C) after longer ageing time (3000 hours) lead to a low impact toughness and brittle fractures both locally and on a macro-level in the specimen. The nickel base alloy show higher impact toughness with increasing ageing temperature and time. Local approach to fracture should be applied by considering the heterogeneous mechanical behaviours in these two kinds of materials.

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