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Ferritic phase transformation to improve creep properties of martensitic high Cr steels



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ABSTRACT

Creep properties of two 9% Cr steels, heat treated based on a quenching and partitioning concept are investigated. Austenite is partly transformed into martensite during interrupted quenching. Due to the new interfaces and the deformed retained austenite, it is possible to transform the retained austenite into ferrite and precipitates during subsequent tempering. The kinetics of this transformation is described by the Kolmogorov-Johnson-Mehl-Avrami equation. Creep rupture times are improved by more than 50% compared to conventional heat treated samples. It is expected that this heat treatment improves the creep properties of the entire class of 8–13% Cr steels.

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Quality heat treatment of steels offers the possibility to adjust their microstructure and related mechanical properties in application [1]. The conventional heat treatment for 8–13% Cr martensitic steels is austenitizing followed by quenching below martensite finish (M_f) temperature and tempering (Q&T). During cooling from the austenitizing temperature, the transformation behaviour of these steels is predominantly influenced by alloying elements. Additions of e.g. Cr, Mo, Mn, Ni, C and ppm of B increase hardenability by delaying the reconstructive transformation of austenite (γ) to ferrite (α) [2,3], which starts below A_{r3} temperature. This leads to a complete martensitic morphology upon air cooling to room temperature for section thicknesses below approximately 100 mm. The crystallographic relationship between the parent γ and the transformed martensite (α') is described by a mixed Kurdjumov-Sachs, Nishiyama-Wassermann orientation-relationship with 24 possible variants [4–7]. The typical microstructure of these steels after the Q&T heat treatment is therefore tempered martensite with precipitates predominantly nucleated at the prior austenite grain boundaries (PAGBs), as well as at the packet, block and martensite lath boundaries [8].

For application at high temperatures, the thermal stability of the precipitates along these boundaries has been found to have a major influence on the creep rupture life of these steels [9–11]. Therefore, a lot of effort was spent for the fine tuning the alloying composition with

emphasis on precipitate forming elements to increase their number density and phase fraction along these boundaries [12–15]. Coarsening of precipitates leads to the loss of boundary pinning forces and to coarsening of the block and lath boundaries, which is regarded as the creep strain rate ($\dot{\epsilon}$) governing process in tertiary creep [9,16,17]. In the secondary creep regime at $\dot{\epsilon} < 1e - 10 [s^{-1}]$, $\dot{\epsilon}$ is predominantly governed by diffusion whereas at $\dot{\epsilon} > 1e - 10 [s^{-1}]$ dislocation creep governs the $\dot{\epsilon}$ [17–21]. In this regime, a lower $\dot{\epsilon}$ can be achieved by a lower diffusion coefficient as well as by a higher number density and stability of precipitates.

In this work, the quenching and partitioning (Q&P) heat-treatment concept of [22] is adopted for 8–13% Cr heat resistant steels. The corresponding phase transformations are investigated using a Bähr 805 A/D dilatometer [23]. The lever rule is applied to evaluate the amount of isothermally transformed γ at different temperatures. By measuring the length of the vertical lever at three temperatures; before and after the complete α to γ transformation (at A_{c1} and A_{c3} temperature); at the martensite start (M_s) point, the length of the lever is developed temperature dependent in the range between M_s and A_{c1} temperature. At these three temperatures the total transformed phase fraction (f) is set to 95%. The reduction of the lever by the superposition of the relaxing α' with the growing α during tempering was neglected due to low contribution and the transformed f is calculated with the Kolmogorov-Johnson-Mehl-Avrami (KJMA) equation [24]. The calculated transformed amounts are indicated into a time temperature transformation (TTT) diagram [25]. Q&P and Q&T are carried out on two state of the art 9%Cr steels for comparative creep rupture tests at 923 K and 150 MPa. The

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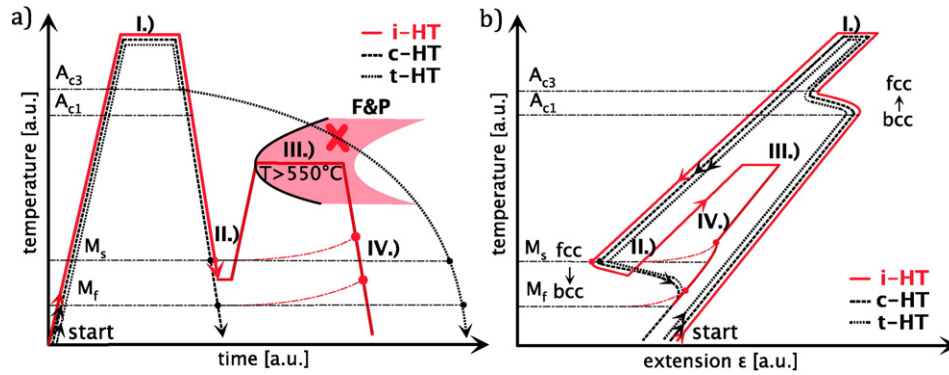


Fig. 1. Accessibility of the $\gamma \rightarrow$ F&P transformation with a) Three performed heat treatments, and b) Corresponding qualitative dilatometry curves for the HT cycles.

development to today's state of the art took decades of research and is proposed to the standards to be Grade 93 [26–30].

Three different heat treatments, i.e. conventional heat treatment (c-HT), theoretical heat treatment (t-HT) and improved heat treatment (i-HT) are illustrated in Fig. 1a) with the expected dilatometer curves in Fig. 1b). All three heat treatments start with solution annealing after complete austenitization above A_{c3} temperature (step I.). The indicated c-HT follows the rules of the mentioned Q&T process (step II.). The theoretical heat treatment (t-HT) is slow cooling and illustrates the transformation of austenite into ferrite and precipitates (F&P in Fig. 1a)) is not accessible within process relevant times, because martensite (α') formation takes place at cooling rates even lower than 1 K min^{-1} . The i-HT aims to cool the steel between M_s and M_f in order to partially transform the austenite into α' and then to directly heat to the tempering temperature between 873 K and 1073 K (step III.) during which the deformed retained austenite (RA) transforms into ferrite and precipitates (F&P) while α' is tempered. Depending on the tempering conditions, RA (if any) transforms into α' (step IV.) at a higher M_s temperature than during the initial cooling (step II.). The i-HT is also investigated on a 13%Cr steel with laser-scanning confocal microscopy and can be watched live [31].

For this study the chemical composition of the investigated steels is provided in Table 1. Their compositions mainly differ from the C, Co, W and Mo content.

The applied austenitization temperature is 1393 K for 30 min followed by quenching to a temperature of 633 K for T516 and 603 K for T570. At these temperatures, the corresponding phase fraction of RA is 30% for the T516, 75% for the T570 steel and is indicated in Fig. 2a) and b). The amount of transformed RA is proportional to the increasing lever during subsequent isothermal tempering (see step III.) in Fig. 1b)) between 873 K and 1073 K for 3 h. The evolution of the f during this time- and temperature dependent transformation is described with the parameters of the KJMA equation [24]. The parameter C is diffusion (i.e. composition, temperature and time) dependent, while the coefficient n is temperature independent and can be used for a rough categorization of growth [32]. For temperatures between 923 K and 1023 K, the evaluation of n shows mean values of $n_{T516} = 1.4 \pm 0.2$ and $n_{T570} = 1.1 \pm 0.1$. These n values refer to nucleation at the grain boundary or grain edge, growth of particles of appreciable initial volume or needles and plates of finite long dimensions which are small in comparison with their separations and to thickening of long cylinders [32]. At temperatures of

1073 K and 873 K the RA to ferrite transformation was not detected within 3 h. Therefore, the transformation of RA into α' occurs during the cooling step IV.) (in Fig. 1 b)).

$$\ln(1-f) = -Ct^n \quad (1)$$

The time required to achieve a specific relative amount of transformed RA into F&P can be viewed in the TTT-diagram in Fig. 2c). The C-shaped curves show that the reaction kinetic is quite similar for the two steels despite their different initial amounts of α' and their different chemical composition. To be able to draw the 10%, 50% or 90% lines, the relative achievable amount for the transformation (i.e. RA) is scaled to 100% and the lines are calculated using Eq. (1).

As a next step, comparative creep tests were carried out for both materials at 923 K and 150 MPa for with a specimen diameter of 7.07 mm. Prior to creep testing, the specimens for T516 and T570 were austenitized at 1373 K and 1423 K for 30 min and 180 min respectively to produce prior austenite grain sizes (PAGS) between 50 μm and 700 μm . Preliminary dilatometry tests did not show a systematic influence of the PAGS on the M_s temperature [33]. One series of creep specimens was treated with the i-HT and quenched to 633 K (T516) and to 593 K (T570), after which it was tempered twice at 973 K for 3 h. The other series was conventionally quenched below M_f (c-HT) and, similar to the first series, was tempered twice. The creep rupture times (t_r) from the comparative uniaxial creep tests are presented in Fig. 3a) for t_r -times ranging from 1000 to 13,000 h. T516 shows creep rupture times six times longer than those of T570. The superior creep rupture time of the i-HT is observed for both steels at each PAGS. Comparing c-HT with i-HT, the combination of tempered martensite with the ferritic phase transformation improves the creep rupture times, on average, by more than 50%. With increasing creep exposure time, the difference becomes more significant. A PAGS of 300 μm provides the best creep results for both steels [34], despite premature intergranular failure of the specimen T516 c-HT due to a surface defect. The decrease of creep rupture times as PAGS decrease is most likely due to the higher amount of interfaces [35] promoting diffusional processes and an increase of recovered zones [36]. Increasing the PAGS leads to a change in the creep rupture mechanism from ductile to intergranular brittle, with no reduction of area at a PAGS of 700 μm . In comparison to PAGS 300 μm , the larger boundary area in combination, with the high concentration of voids and segregating elements (e.g. P, S) [37] seem to orient the

Table 1

Transformation temperatures (heating- and cooling rate 1 K/s for A_{c1} , A_{c3} and $t_{8/5} = 3 \text{ min}$ for M_s) with the chemical composition of the investigated steels in wt.%.

#	name	A_{c1} [K]	A_{c3} [K]	M_s [K]	C	Mn	Cr	Mo	Co	W	V	Nb	N	B
T570	FB2-2-LN	1138.5	1171.7	628.2	0.16	0.3	8.8	1.4	1.0		0.2	0.04	0.01	0.015
T516	NPM1-LN	1146.6	1202.4	678.0	0.07	0.5	9.0		2.9	2.9	0.2	0.05	0.01	0.01

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