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High-temperature mechanical properties improvement on modified 9Cr–1Mo martensitic steel through thermomechanical treatments

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ABSTRACT

In the framework of the development of generation IV nuclear reactors and fusion nuclear reactors, materials with an improved high temperature (≅650 °C) mechanical strength are required for specific components. The 9–12%Cr martensitic steels are candidate for these applications. Thermomechanical treatments including normalisation at elevated temperature (1150 °C), followed by warm-rolling in metastable austenitic phase and tempering, have been applied on the commercial Grade 91 martensitic steel in order to refine its microstructure and to improve its precipitation state. The temperature of the warm-rolling was set at 600 °C, and those of the tempering heat-treatment at 650 °C and 700 °C thanks to MatCalc software calculations. Microstructural observations proved that the warm-rolling and the following tempering heat-treatment lead to a finer martensitic microstructure pinned with numerous small carbide and nitride particles. The hardness values of thermomechanically treated Grade 91 steel are higher than those of the as-received Grade 91. It is also shown that the yield stress and the ductility of the thermomechanically treated Grade 91 steel are significantly improved compared to the as-received material. Preliminary creep results showed that these thermomechanical treatments improve the creep lifetime by at least a factor 14.

1. Introduction

The development of generation IV nuclear reactors and fusion nuclear reactors requires the use of materials able to work at higher temperatures (up to 650 °C) compared to the service conditions of the present generations of reactors [1].

9–12%Cr martensitic steels are widely used for conventional energy production and have thus been chemically optimised for creep resistance. They are now candidate materials to replace austenitic stainless steels in future nuclear reactors. Their main advantages compared to austenitic stainless steels are a lower thermal expansion, a better thermal conductivity, a lower material cost and a good resistance to stress corrosion. These steels are hardened by several populations of precipitates (such as MX nitrides and carbides, $M_{23}C_6$ carbides ...). The nitrogen and carbon contents as well as the austenitisation temperature have a direct effect on the precipitates characteristics [2].

This study is devoted to materials for components like heat exchanger, hot gas ducts, vapour generator, etc. Since these components will be subjected to high-temperature cyclic loadings, data about creep–fatigue properties are required in design procedures. Recent studies on commercial Grade 91 ("G91" which is a modified 9Cr–1Mo steel) showed that cyclic loadings coupled to high-temperature creep loadings lead to a fast and strong cyclic softening effect, which deteriorates the mechanical properties such as the creep strength [3–7]. This phenomenon was shown to be mainly due to a coarsening of the microstructure, in particular the decrease of the dislocation density, and the coalescence of precipitates.

Warm deformation of metastable austenite in high strength steels was shown in the 1960s to lead to a significant hardening of martensite. This effect is used in the "ausforming" thermomechanical treatment [8–10]. For example a study dealing with a 13%Cr, 0.3%C steel showed that the ausforming treatment modifies the tempering stages of martensite and allows a finer precipitation of carbides [11]. Recent works suggest that thermomechanical treatments (TMT) applied to 9–12%Cr steels could improve their mechanical strength by refining the precipitation state, either by cold-rolling [12,13] or warm-rolling [14,15].

The purpose of the present study is to apply this kind of thermomechanical treatment on steel G91: firstly to define the best parameters for TMT on G91, and secondly to test the mechanical influence of this treatment on the high temperature deformation and damage mechanisms of this material. The influence of the rolling conditions with regard to that of the austenitisation and tempering temperatures are discussed.

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These TMT parameters were investigated using the MatCalc software [16] and the most relevant conditions were selected. Then the material was thermomechanically treated and the as-quenched warm-rolled G91 was studied in terms of microstructure by optical and electron microscopy. Microhardness measurements and thermo-electrical power (TEP) measurements were performed in order to choose the best tempering temperatures. The material was then tempered and the effects of the whole treatment on the microstructure and the mechanical strength were investigated.

Results are presented in a chronologic way in order to highlight the methodology.

2. Materials and experimental procedures

2.1. Base material: G91 steel

The base material under study in this article is the commercial G91 martensitic steel. Its composition is given in Table 1. The steel is used under the form of small blanks (100 mm × 40 mm × 30 mm) that were cut longitudinally from a large plate (2000 mm × 1000 mm × 30 mm).

The as-received material (AR) was normalised at 1050 °C for 30 min, then air quenched, and tempered for 1 h at 780 °C. This material exhibits a martensitic microstructure with several scales (Figs. 1 and 2) [2]:

- the former austenitic grains formed during the normalising heat-treatment (20–25 μm),
- in each grain there is one or several packets of laths, subdivided into blocks of laths (that have the same {1 1 1}γ plane),
- the blocks are made of 5–10 parallel martensitic laths (width 0.25–0.5 μm),
- each lath is composed of subgrains formed during the tempering heat-treatment (0.25–0.5 μm).

The dislocation density of G91-AR is about 1.1–1.6 × 10^{14} m^{-2} [4]. Different kinds of precipitates (see Fig. 2) prevent their motion [5–7,12,14,18,19]:

- $M_{23}C_6$: around 100–120 nm, located on prior austenitic grain boundaries,
- MX: small nitrides and carbides of Nb or V, around 30–50 nm, distributed homogeneously within the matrix. They are very stable in temperature [19]. These precipitates pin the dislocations and the subgrains boundaries. A few MX particles have a particular V-Wing composed of a Nb-rich particle with V-rich wings (Fig. 2b) [17].

2.2. Thermomechanical treatment

The purpose of the TMT investigated in the present study is to reinforce the pinning mechanism of the dislocations by the MX particles in order to increase the mechanical strength of G91 steel. Warm-rolling in austenitic phase is expected to introduce a high dislocation density. Therefore the martensitic transformation should be modified: the martensite laths and subgrains should be refined and the higher dislocation density should allow a more homogeneous distribution of particles.

The principle of the thermomechanical treatment including warm-rolling is presented in Fig. 3. After a second austenitisation at 1150 °C (see below), the steel is air-cooled (−1 °C/s) to the rolling temperature. The total duration of the warm-rolling step is about 10 min. The reduction in thickness by warm-rolling is 25% and the rolling is carried out in three passes. Warm-rolling is followed by water quenching.

Some of the TMT parameters are investigated first and then fixed (i.e. the heating and cooling rates, the rolling conditions – see Section 3); others like the temperatures and durations of the different steps are kept free and are chosen thanks to simulations using the MatCalc software.

Vickers microhardness measurements (500 g) were performed, and thermo-electrical power (TEP) measurements were made on the rolled and quenched material (itself and also submitted to different tempering heat-treatments).

Extractive carbon replicas were prepared by etching the polished steel surface 10 min in a solution of 1% tetramethylammonium chloride and 10% acetylacetone in methanol, followed by the evaporation of carbon onto the etched steel sample, then dissolution of the matrix in the same solution at a voltage of 1.2 V. These carbon replicas as well as thin foils were examined using a JEOL-2010FEG transmission electron microscope, for both quenched and tempered warm-rolled materials.

Creep tests were performed on the tempered warm-rolled materials at 650 °C under 120 MPa. Tensile tests were carried out at a strain rate of 7 × 10^{-4} s^{-1}, at 20, 550 and 650 °C.

3. Results and discussion

3.1. MatCalc simulations

The MatCalc software (developed by Ernst Kozeschnik from TU Graz [16]) was used in order to simulate the precipitation kinetics during the thermomechanical treatment. The effect of several heat-treatments on the characteristics of MX and $M_{23}C_6$ precipitates were studied.

The values of the fixed parameters given to the software are presented in Table 2. MatCalc needs these values for simulating the precipitation state and the user must fix them. However it has to be noticed that the dislocations densities, mainly those of austenite at 1150 °C, are not precisely known and the values are estimated.

The temperature and duration of each step are chosen as follows:

- The temperature of the second normalisation treatment is chosen at 1150 °C in order to dissolve all the $M_{23}C_6$ carbides and as much MX carbides and nitrides as possible, while maintaining the same prior austenitic grain size. The VN will indeed dissolve almost entirely in 1 h at 1150 °C [18]; however the Nb(C, N) particles will not dissolve at all, according to Klueh and Harris rules [20].
- Temperature of the warm-rolling must stay above the martensitic start temperature, avoid the ferrite stability domain [2], and be low enough to avoid dislocation annihilation by re-crystallization or recovery. Considering all these constraints, warm-rolling temperature could be investigated (with MatCalc) between 600 °C and 700 °C, and around 800 °C.

Table 1
Chemical composition in wt.% of Grade 91 steel.

C	Cr	N	Mn	Mo	Si	Nb	V	Cu	Ni	S	P	Al
0.088	8.91	0.04	0.363	0.917	0.324	0.08	0.198	0.068	0.15	0.001	0.017	0.018

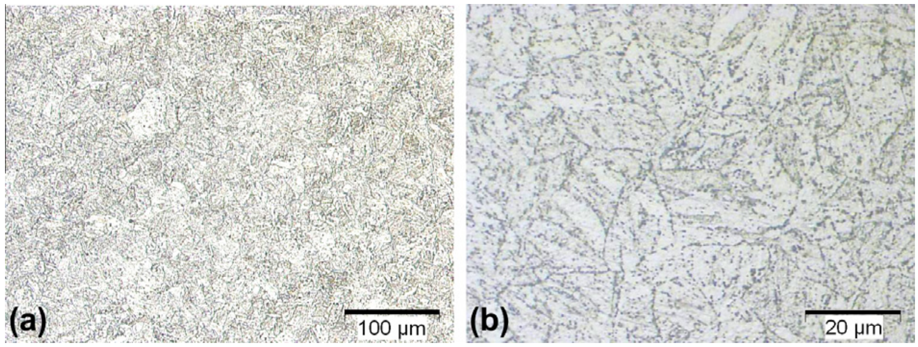

Fig. 1. Optical microscopy observations in the as-received state (G91-AR) showing prior austenitic grains (a); blocks and packets (b).

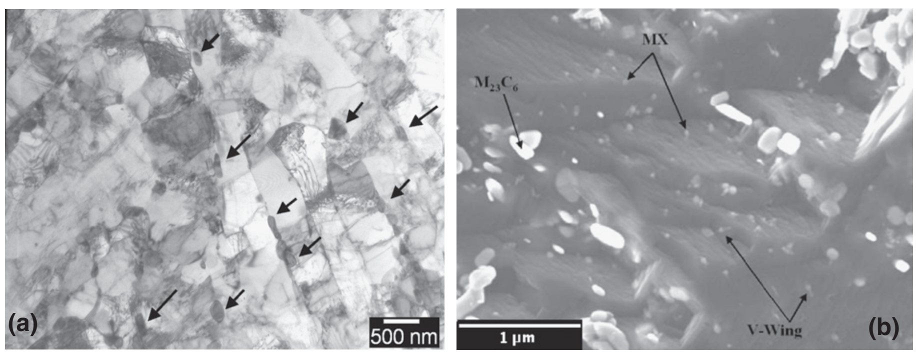

Fig. 2. (a) Bright field TEM observation of G91-AR showing laths, subgrains, precipitates and dislocations (arrows show $M_{23}C_6$ precipitates) and (b) extractive carbon replica observation in SEM-FEG [17].

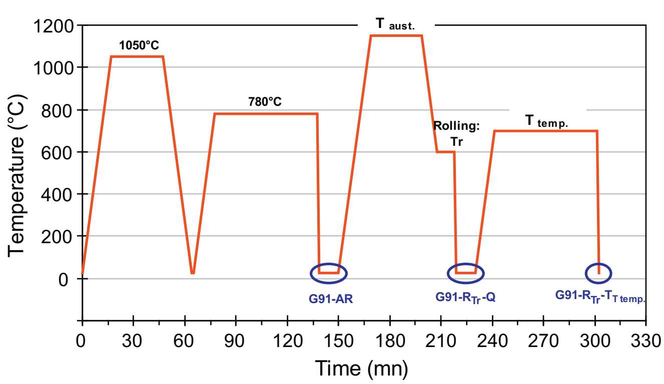

Fig. 3. Thermal cycles of the first heat-treatment and of the thermomechanical treatment under study. The different states of the material are denoted: AR = as-received, R = rolled, Q = quenched, T = tempered.

– Several temperatures of tempering can be simulated; the simulation results as well as the TEP measurements provide further information for the final choice. Anyway the tempering temperature has to be at least equal to 650 °C, which is the expected service temperature for this material.

Fig. 4 shows an example of the results obtained after a MatCalc simulation.

The simulation of the G91-AR shows that MatCalc underestimates the size of the precipitates and overestimates their density; however it gives a correct order of magnitude as shown in Table 3.

The precipitation rates given by the calculation are not realistic since they appear to be very high. In fact MatCalc calculations were used to compare between two thermomechanical treatments but not to predict absolute size and density.

Table 2
Parameters of each step used for MatCalc calculations.

	Austenite (1st and 2nd austenitisation)	Martensite after 1st quenching	Austenite after warm-rolling	Martensite after warm-rolling and quenching
Grain size (μm)	40	Subgrain 0.4	40	Subgrain 0.4
Dislocation density (m^{-2})	10^{11}	10^{14}	10^{12}	5×10^{15}

The results of several simulations are summarized in Table 4. Small hardening MX precipitates will mainly be discussed because they are of first interest for the mechanical properties [7,19].

3.1.1. Austenitisation

As expected, the whole fraction of MX precipitates is not dissolved during the second normalisation treatment. MatCalc results show that these remaining MX precipitates grow up during the following air quenching (≅−1 °C/s).

3.1.2. Warm-rolling

There is no significant difference between MX density after warm-rolling at 600 °C and at 700 °C as no further precipitation occurs during warm-rolling. However it is expected that recovery will be more pronounced at 700 °C than at 600 °C, which implies a lower density of dislocations, whereas the purpose of the warm-rolling step is to increase it. Furthermore, the temperature of 700 °C is close to the ferrite stability domain, thus warm-rolling temperature was selected at 600 °C.

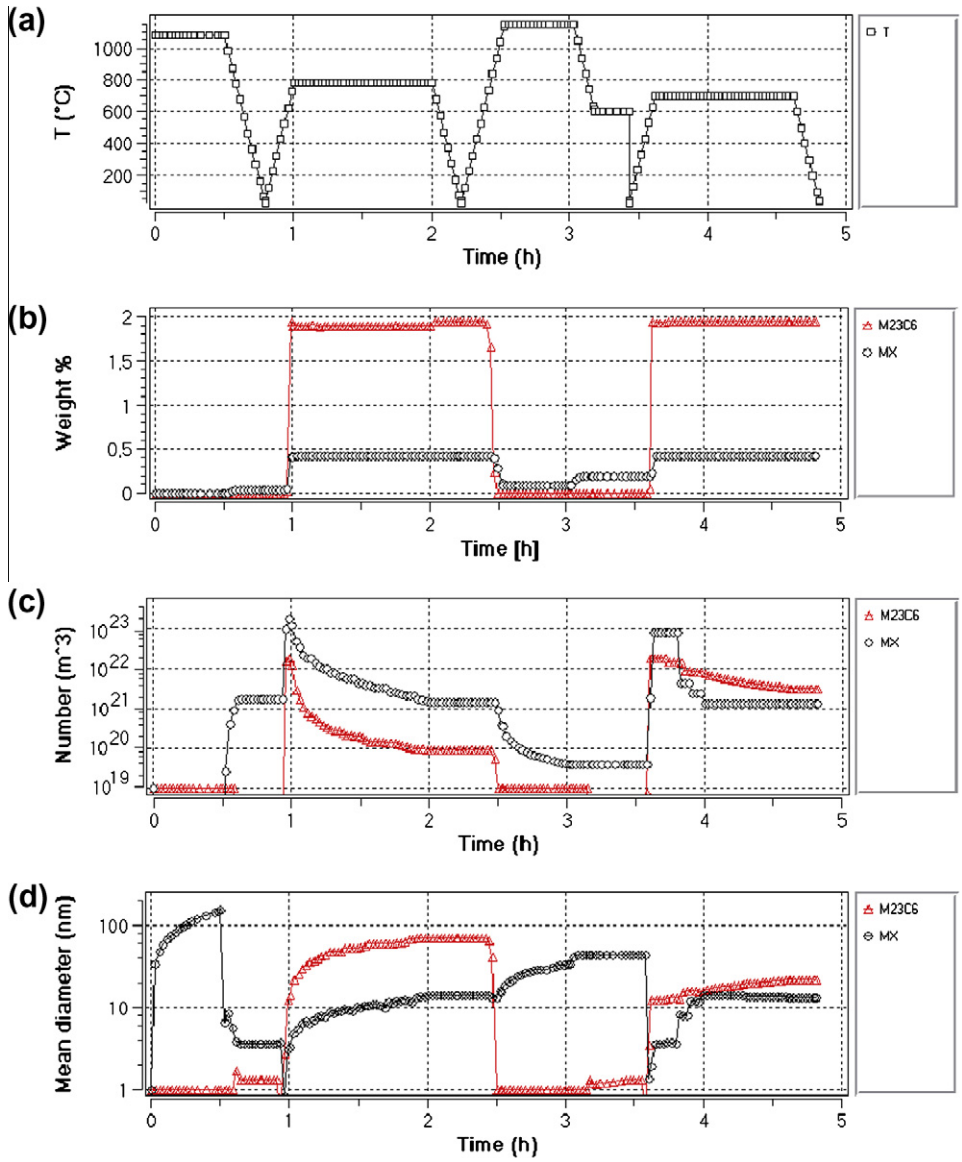

Fig. 4. MatCalc simulation of the precipitate populations obtained with a warm-rolling treatment (10 min, 600 °C) followed by a tempering (1 h, 700 °C).

Table 3
Comparison between observation and MatCalc simulations of MX and $M_{23}C_6$ precipitates for the G91-AR steel (normalised at 1050 °C, tempered at 780 °C).

Precipitates	Experimental diameters [16,22]	MatCalc simulation results (nm)
MX	30–50 nm	14
$M_{23}C_6$	≅100 nm (largest ones, disc-or needle-shaped :≅300 nm at grains and packets boundaries; smallest ones: 50–80 nm at subgrains boundaries)	71

3.1.3. Tempering

Most of the MX precipitates are formed during the tempering treatment. For one rolling temperature, a finer population of MX is obtained after 1 h at 650 °C than after 1 h at 700 °C.

Other calculations have been made; they show that the properties of the precipitates (small size, high density) are improved with the decreasing tempering temperature. This could lead to choose a low annealing temperature. Nevertheless the temperature and time of tempering have to be high enough to ensure a suitable dislocation density. Indeed MatCalc does not calculate the final dislocation density; it is a parameter estimated by the user. But the dislocation density has a role on the mechanical properties, and one of the interests of the tempering is to soften the microstructure to improve impact properties.

According to MatCalc, after 1 h at 650 °C almost all the MX have formed (i.e. 0.421 wt.% – the equilibrium fraction is 0.427 wt.%); and after 1 h at 700 °C the whole fraction is formed. Therefore these two temperatures give a good compromise between the complete precipitation of the MX, and a high density of small MX precipitates.

For these reasons it has been decided to test a thermomechanical treatment based on a warm-rolling at 600 °C. MatCalc simulations suggest that the temperature of the tempering treatment should be between 650 °C and 700 °C. This choice will be validated by thermo-electrical power and microhardness measurements (see Section 3.3).

3.2. Microstructure of the rolled and quenched G91: G91-R600-Q

Vickers hardness measurements have been performed on the warm-rolled steel sample. Its hardness is about 470 Hv. This value

Table 4
Characteristics of the precipitates populations in Grade 91 steel after simulated thermomechanical treatments (italics letters are for rolled and quenched materials).

Notations	$M_{23}C_6$			MX		
	$\%_w$	Number/m^3	Mean diameter (nm)	$\%_w$	Number/m^3	Mean diameter (nm)
Initial "G91-AR"	1.947	9.5×10^{19}	71	0.427	1.47×10^{21}	14
Warm-rolling 600 °C "G91-R600-Q"	*0*	*n.a.*	*n.a.*	*0.195*	3.97×10^{19}	*44*
Warm-rolling 600 °C +1 h tempering 650 °C "G91-R600-T650"	1.955	1.86×10^{22}	12.5	0.421	9.1×10^{22}	3.5
Warm-rolling 600 °C +1 h tempering 700 °C "G91-R600-T700"	1.955	3.15×10^{21}	22	0.427	1.36×10^{21}	13.5
Warm-rolling 700 °C "G91-R700-Q"	*0*	*n.a.*	*n.a.*	*0.195*	4.6×10^{19}	*38*
Warm-rolling 700 °C +1 h tempering 700 °C G91-R700-T700	1.955	3.23×10^{21}	21.7	0.427	10^{21}	16.5

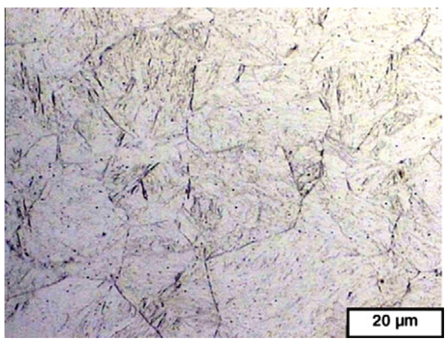

Fig. 5. Optical microscopy observations of the G91-R600-Q steel in the transverse direction.

is higher than that of quenched reference Grade 91 steel (around 410–420 Hv for a normalisation heat-treatment between 1050 °C and 1160 °C [2]).

This increase in hardness, due to ausforming, is related to the refinement of the martensitic microstructure and to a possible increase in the dislocation density.

The sample G91-R600-Q exhibits a martensitic microstructure (Figs. 5 and 7a) with still some precipitates. Transmission electron microscopy (Fig. 6b) and scanning electron microscopy on a carbon replica (Fig. 6) show small precipitates containing Nb. This is consistent with the predictions of Klueh and Harries [20]. Their sizes are included between 10 nm and 150 nm; 90% of the precipitates are smaller than 50 nm in diameter; these values correspond to MX diameters.

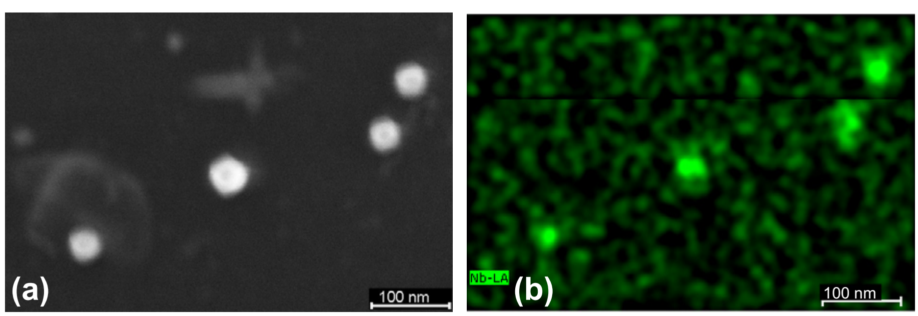

Fig. 6. (a) SEM-FEG observation of a carbon replica of G91-R600-Q and (b) associated EDS mapping of the Nb content.

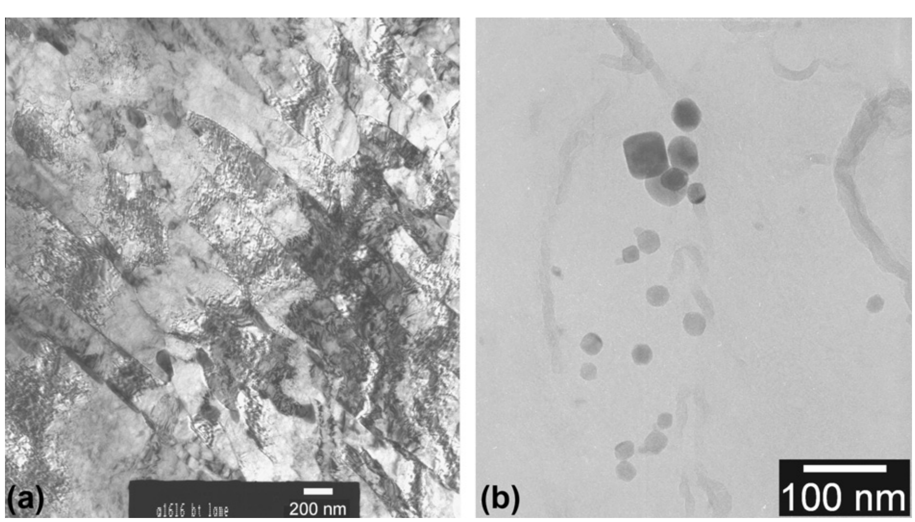

Fig. 7. TEM observations of the G91-R600-Q: (a) thin foil showing laths and (b) carbon replica showing precipitates.

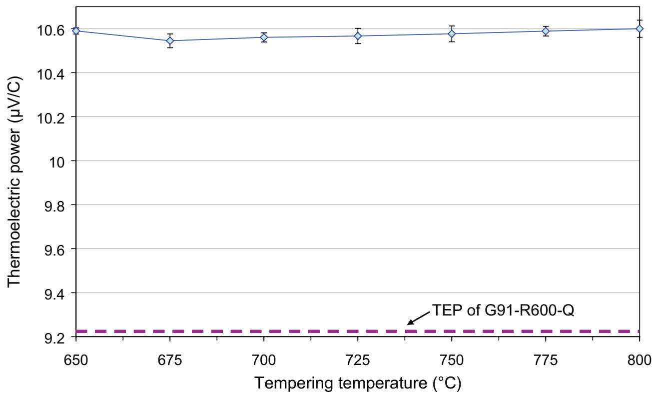

Fig. 8. Thermo-electrical power versus temperature of annealing treatments (1 h) on the G91-R600-Q.

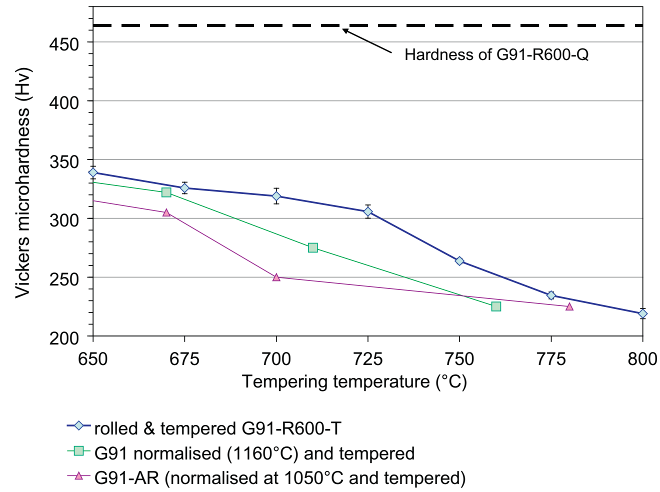

Fig. 9. Vickers hardness versus temperature of annealing treatments (1 h) on the G91-R600-Q.

The absence of vanadium precipitates and the weak density of niobium precipitates prove that the normalisation treatment at 1150 °C has dissolved not only all the VN nitrides and the VC and $M_{23}C_6$ carbides, but also some of the Nb(C, N) carbonitrides.

MatCalc does not predict the precipitation of MX during the rolling step, and the MX precipitates of Fig. 7b might be some NbC precipitates that were not dissolved during the austenitisation.

3.3. Selection of the tempering temperature

Tempering tests of 1 h were performed every 25 °C between 650 °C and 800 °C on the G91-R600-Q material. Thermo-electrical power of each tempered sample was measured. The TEP value of the quenched G91-R600-Q is 9.20 $\mu V\,K^{-1}$. For the tempered samples it increases with temperature, from 10.54 to 10.60 $\mu V\,K^{-1}$ (see Fig. 8).

This method is sensitive mainly to solid solution elements C and N, but also to the dislocation density and to coherent precipitates (i.e. MX) [21]. Therefore the $M_{23}C_6$ carbides only have an effect due to the subsequent decrease of solid solution elements, while the MX particles have a similar effect and also an additional effect. However the weight fraction of MX precipitates is weak and is not expected to have a very significant impact on the measure.

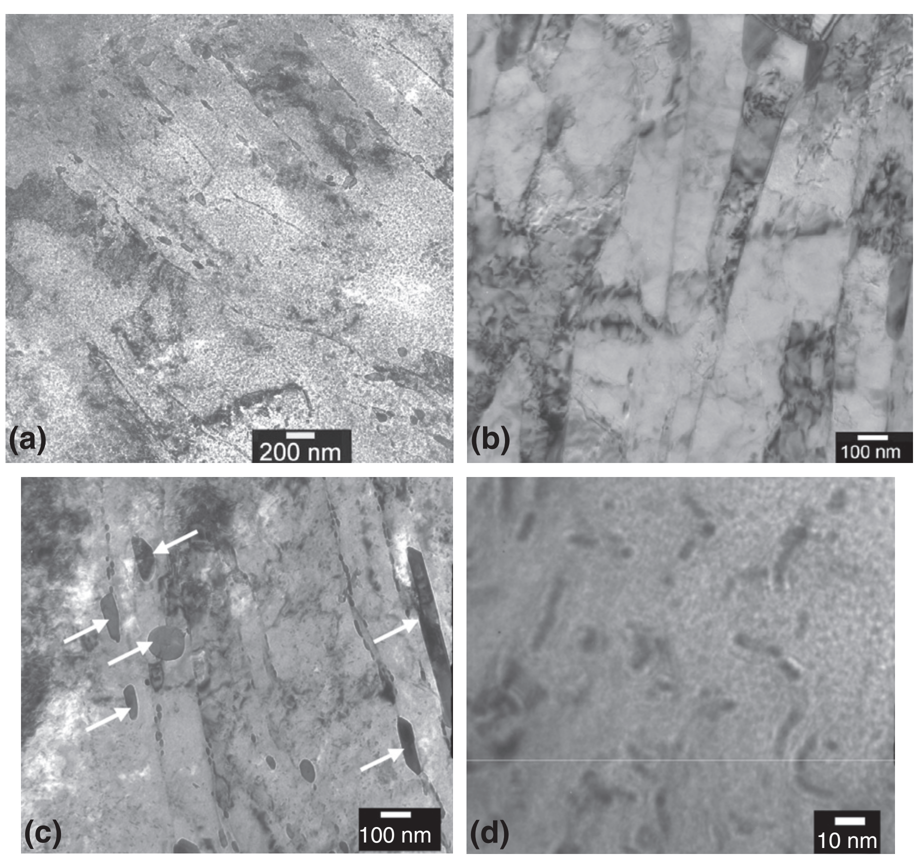

Fig. 10. Bright field TEM micrographs on thin foils of : (a) G91-R600-T650 laths, (b) G91-R600-T700 laths, (c) $M_{23}C_6$ precipitates and (d) small MX precipitates in the subgrains.

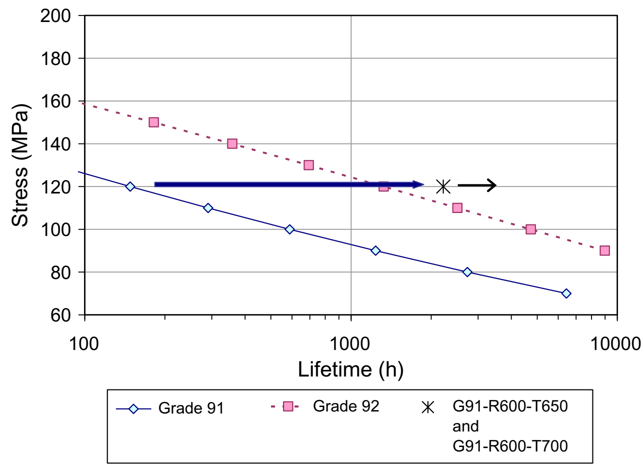

Fig. 11. Creep lifetimes versus applied stress at 650 °C of G91-R600-T650 and of G91-R600-T700 compared to G91-AR [24] and P92-AR [24].

The increase of the TEP value, from 9.20 to 10.54 μV K^{-1} is due to the precipitation of carbides and nitrides and to the decrease of the dislocation density. A slight increase with temperature occurs between 650 °C and 800 °C, which might be due to the end of the precipitation of the MX particles (0.421 wt.% after 1 h at 650 °C, 0.427 wt.% after 1 h from 700 °C) and to a further decrease of the dislocation density.

Microhardness values of rolled and tempered Grade 91 material decrease with the increasing tempering temperature, slightly between 650 °C and 725 °C and more rapidly between 725 °C and 800 °C (Fig. 9). The most pronounced hardening effect compared to the conventional tempered reference G91 steel is found for temperatures around 700–725 °C. This produces a kind of secondary hardening effect in the warm-rolled material compared to the AR steel.

MatCalc simulations showed that 1 h at 700 °C is sufficient to precipitate the whole weight fraction of MX and of $M_{23}C_6$, and after 1 h at 650 °C all the $M_{23}C_6$ carbides and almost all the MX are formed. In order to have a good compromise between the mechanical strength and the characteristics of the precipitates population, temperatures of 650 °C and 700 °C have been selected for the tempering treatment.

3.4. Microstructure of the rolled and tempered G91: G91-R600-T650 and G91-R600-T700

The microstructures of both rolled and tempered G91 (Fig. 10a and b) are finer than that of G91-AR; it is worth noting that the laths width of G91-R600-T700 is 1.7 times smaller (i.e. a mean width value of 210 nm, while the mean laths width of G91-AR is 370 nm [22]).

TEM micrographs on thin foils show three kinds of precipitates. Relatively large $M_{23}C_6$ carbides (200 nm) are observed mainly at triple boundaries and along laths boundaries (Fig. 10c); some are even a few hundreds nanometers long. Intermediate size precipitates have also formed on the laths boundaries (around 100 nm), these precipitates are $M_{23}C_6$ too. $M_{23}C_6$ sizes are close to that of the $M_{23}C_6$ usually found in G91-AR.

Inside and along the laths small needle-shaped precipitates have formed. Their mean size is 5 nm × 11 nm. These values are smaller than those of the MX particles found in G91-AR (30 nm × 50 nm).

3.5. Tensile and creep tests results

Creep tests (120 MPa, 650 °C) are being carried out on both G91-R600-T650 and G91-R600-T700. These tests are still running but their duration can be compared to those of the reference material G91-AR (see Fig. 11).

These results show that thermomechanical treatments lead to an increase of the creep lifetime at 650 °C under 120 MPa: lifetimes are at least 14 times larger than that of G91-AR, and also larger than that of the Grade 92.

These creep preliminary results are not comparable to the creep lifetimes of the modified 9Cr–1Mo steel treated by Klueh [14] since this author has carried out creep tests on a steel that was rolled and shortly annealed before martensitic transformation, but not tempered afterwards as in the present study.

Tensile tests (Fig. 12) show that the thermomechanically treated materials exhibit much higher yield strengths (increase of 360 and 430 MPa at 20 °C; 200 and 300 MPa at 550 °C) and a better ductility than the as-received G91 material (Table 5). However at high temperatures, the tensile curves show the work-softening of the optimised materials.

At 20 and 550 °C, the behaviour of the G91-R600-T650 is better than that of G91-R600-T700 whereas this is the contrary at 650 °C. These effects can be correlated to the fact that the testing temperature, 650 °C, is also the annealing temperature of the G91-R600-T650 steel. Therefore, at 20 °C and 550 °C the finer population of precipitates of the G91-R600-T650 (compared to the G91-R600-T700) gives a better mechanical strength, while some microstructural evolutions still take place during the tensile tests at 650 °C. This is confirmed by the very noisy shape of the creep curve of the G91-R600-T650 compared to the G91-R600-T700.

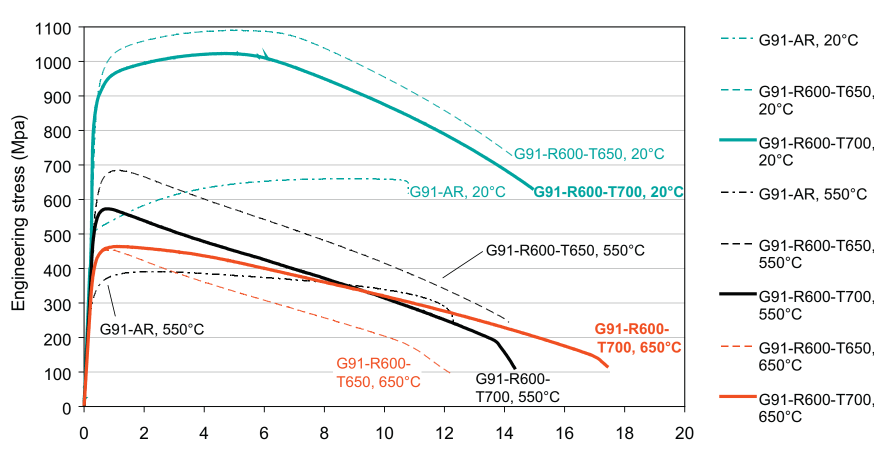

Fig. 12. Tensile tests results of G91-R600-T650 and of G91-R600-T700 compared to G91-AR.

Table 5
Tensile properties of G91-R600-T650 and G91-R600-T700 steels compared to G91-AR steel.

	G91-AR	G91-AR	G91-R600-T650	G91-R600-T650	G91-R600-T650	G91-R600-T700	G91-R600-T700	G91-R600-T700
Temp. (°C)	20	550	20	550	650	20	550	650
R_m (MPa)	660	390	1090	684	454	1023	573	464
$R_{p\ 0.2}$ (MPa)	520	390	980	655	438	931	564	443
Ar (%)	10	12	13.2	13.7	12	14	14.2	17

In comparison, Klueh's tensile tests results on a modified 9Cr–1Mo steel, once as-received and once thermomechanically treated and tempered, show a loss of 45 MPa at 20 °C, a gain of 20 MPa at 550 °C and 55 MPa at 650 °C [23].

4. Conclusions

Thermomechanical treatments, including warm-rolling in metastable austenitic phase, have been carried out on a commercial modified 9Cr–1Mo steel. Their conditions have been adjusted using MatCalc simulations.

The microstructures of the materials have been investigated by optical and electron microscopy after each step of the treatment. The warm-rolling (at 600 °C, 25% deformation) induces an increase of 65 Hv of the hardness of the quenched warm-rolled G91 compared to the quenched reference G91.

The final warm-rolled tempered G91 exhibits a martensitic microstructure with finer laths and smaller precipitates than the as-received G91.

Mechanical tests have been performed. The warm-rolled tempered G91 samples present a higher microhardness than the reference as-received G91: the hardness gain is 15 Hv for a tempering at 650 °C and 46 Hv at 700 °C (only due to the warm-rolling), and the total gain is 100 Hv compared to the G91-AR (effects of both rolling and temperatures of the TMT).

The ongoing creep tests show an improvement of the creep lifetime by a factor larger than 14. The tensile tests show a significant increase in yield stress and also in ductility.

However optimised materials still present work-softening. Fatigue tests are currently being performed in order to check whether these thermomechanical treatments also lead to a reduced cyclic softening effect.

These thermomechanical treatments will be applied to experimental martensitic steels with optimised chemical compositions.

Acknowledgements

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