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Title:

High Performance Ferrite (HiperFer) – A New Alloy Family

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Summary

The high performance ferritic (HiperFer) stainless steel, which is under development at the Institute of Microstructure and Properties of Materials (IEK-2) at Forschungszentrum Jülich GmbH in Germany, provides a unique combination of creep, thermomechanical fatigue, steam oxidation and wet corrosion resistance. The balanced property profile makes it a high potential candidate for highly flexible, future power technology equipment like conventional back-up power plants, concentrating solar power plants or "Power-2-X" conversion systems. This paper outlines the impact of chemical composition, heat and thermomechanical treatment on mechanical properties (tensile, creep, thermomechanical fatigue and Charpy impact strength) of these novel steels and their welds. An overview on the development status reached and the future development and application potentials will be outlined.

Key Words

ferritic steel, creep, fatigue, Charpy impact strength, application potentials

Introduction

Advanced ferritic-martensitic (AFM) 9-12 wt% Cr steels are limited to application temperatures of 600 - 620 °C [1, 2]. Beyond 620 °C the 9 wt% Cr materials are not applicable, because of limited steam oxidation resistance [2]. Higher chromium steels were developed for application beyond 620 °C, but suffer a sigmoidal drop in creep strength during long-term application [3]. This decrease is caused by the formation of the so called Z-phase - a complex Cr(V, Nb)N - at the expense of strengthening MX particles [4, 5]. An increase in chromium content is mandatory to ensure sufficient steam oxidation resistance [6, 7] up to temperatures of 650 °C. For this reason the development of AFM steels might probably be left at an irresolvable conflict of aims.

The novel high chromium HiperFer [8] fully ferritic steels under development at Forschungszentrum Jülich provide a vital concept for resolving this situation: Strengthening of these steels is accomplished by a combination of solid solution and intermetallic $(Fe,Cr,Si)_2(Nb,W)$ Laves particle strengthening [9]. The creep strength potential of these grades is beyond grade 92 and steam oxidation resistance superior to 12 wt% Cr AFM steels [8, 10]. Furthermore the trial alloys provide favourable thermomechanical fatigue resistance [8, 11]. Because HiperFer does not undergo martensitic transformation (thermo)mechanical processing was considered to be mandatory to compensate lacking short-term dislocation strengthening. This drawback was resolved by changes in chemical composition and tailored heat treatment.

Experimental

Base materials

The chemical compositions of five HiperFer trial steels (17Cr1, 17Cr2, 17Cr4, 17Cr5 and 17Cr8) are given in Table 1. The 17Cr1 composition served as the base composition. The 17Cr2 alloy just featured a reduced Mn content. 17Cr4 is a Ni-alloyed variant for increased Charpy impact strength, 17Cr5 a W- and Nb-added version for increased tensile and creep strength on the basis of increased solid solution and particle hardening. 17Cr8 features a low amount of B for increased long-term stability of the strengthening Laves phase particles.

Table 1: Chemical compositions (wt.-%)

	Batch-ID:	C	N	Cr	Mn	Si	Nb	Fe	W	Ni	B
HiperFer	17Cr1	<0.01	<0.01	17.4	0.48	0.24	0.57	R	2.41	-	-
	17Cr2	<0.01	<0.01	17.1	0.18	0.25	0.63	R	2.41	-	-
	17Cr4	<0.01	<0.01	17.0	0.20	0.27	0.61	R	2.53	0.98	-
	17Cr5	<0.01	<0.01	17.2	0.20	0.27	0.99	R	3.70	-	-
	17Cr8	<0.01	<0.01	17.00	0.21	0.29	0.60	R	2.30	-	0.0048

All the materials were produced by vacuum induction melting of high purity elements by the Institute of Ferrous Metallurgy (IEHK), RWTH Aachen University, Germany and rolled to a final plate thickness of 16 mm.
Because changes in chemical composition determine adjusted processing parameters [8] several rolling pass schedules (denoted by "_1" to "_6"), featuring variations in temperature and deformation within the last rolling pass, followed by water quenching, were applied in the production of the plate materials. Parameter sets "_1" to "_3" were applied to the 17Cr1 material only to study the impact of changes in processing [13], set "_4" was applied to trial steels of small chemical variation, (i.e. 17Cr2, 17Cr4 and 17Cr8), while the increased W- and Nb-contents required a specific parameter set "_6" for batch 17Cr5.
Furthermore recrystallization (RX), precipitation (PA) and (simulated) post-weld heat treatment ((sim)PWHT)) studies were carried out mainly at the 17Cr2 material.

Welds

The trial welds intentionally represent the worst-case scenario of a thermomechanically treated, high dislocation density material being welded and later on put into service without any post-weld heat treatment. Microstructural changes, i.e. recovery of excess dislocations or partly recrystallization and with this a drop in dislocation strengthening within the heat affected zone, are inevitable. Founded on this basis optimized base metal heat treatment prior to welding, welding parameters, consumables and post-weld heat treatments are under development.

Trial welds were produced by the Oak Ridge National Laboratory in the US. A couple of as-rolled (i.e. without precipitation heat treatment) 17Cr2_4 plates with 12 mm thickness were manually gas tungsten arc welded (GTAW) by using compositionally matched filler metal strips with 1.6 mm diameter. Total 11 weld beads were filled into a single-V shape groove with pre-heat and inter-pass temperatures in the range from 120 to 150 °C. Visual inspection of the weld surface as well as cross-sectional observation of the weld did not reflect defects related to welding. Some details on weld microstructure can be found elsewhere [12].

Mechanical testing

The tensile experiments were performed applying an electromechanical Instron 1362 testing machine with 10 kN load capability, equipped with a 3-zone resistive furnace and a high temperature extensometer. Strain rates of $10^{-3}s^{-1}$ (at ambient temperature, according to DIN EN 10002-1) and $8{,}33\cdot10^{-5}s^{-1}$ / $8{,}33\cdot10^{-4}s^{-1}$ (elastic / plastic range at elevated temperatures, according to DIN EN 10002-5) were applied.

Dead weight loaded lever arm creep machines, equipped with 3-zone resistive furnaces, were used in creep testing. A temperature accuracy of +/- 1.5 °C was ensured in all the isothermal high temperature experiments by type S thermocouples, attached to the gauge sections of the specimens.

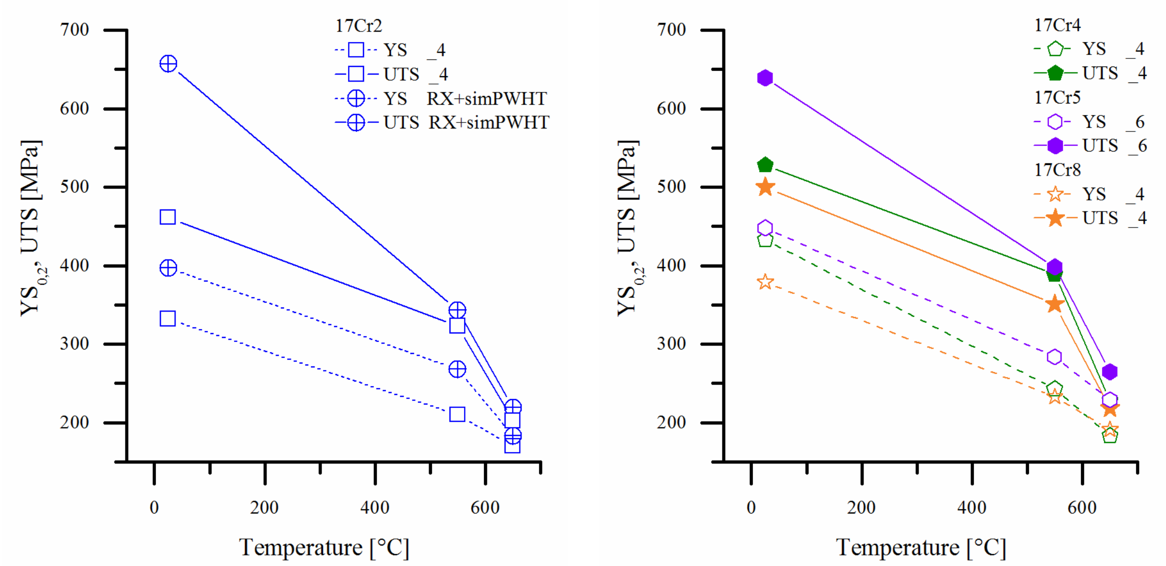
Inductive heating was applied in the thermomechanical fatigue (TMF) experiments. Temperature and strain were controlled by Type R sling thermocouples and extensometers directly attached to the gauge length of the specimens. The out-of-phase (OP) experiments were carried out in total strain control in a temperature range from

50 to 650 °C. Heating/cooling of the specimens was accomplished with a heating/cooling rate of dT/dt = 10 Ks^{-1} without holding time at the hot end of the cycle in order to restrict creep to a minimum. The cooling rate below 200 °C was constricted and thus the cooling cycle took appr. 85 s.

A Zwick 50 J miniature hammer was utilized in impact testing of 27 mm · 3 mm · 4 mm KLST specimens (60 ° notch, 1 mm depth, R = 0.1 mm, distance between anvils: 22 mm). A conversion function was established by comparison of DIN V and KLST impact energy results of solution annealed material, following the procedure outlined by Schill et al. [14].

Results and discussion

Tensile strength



a) b)

Fig. 1: Offset yield and tensile strength of (a) as-rolled ("_4"), recrystallized and post-weld heat treatment simulated ("RX+simPWHT") 17Cr2 and (b) as-rolled 17Cr4_4, 17Cr5_6 and 17Cr8_4 trial steels

Fully ferritic HiperFer steel does not undergo martensitic transformation upon cooling from processing (i.e. rolling or forging). For this reason the intrinsic tensile strength of the as-rolled 17Cr2_4 steel is comparably low (cf. "_4" in Fig. 1a). In [12] it was outlined, that this can be counterbalanced by increased dislocation density through tailored thermomechanical processing. Mechanical properties depending on thermomechanical processing alone might greatly restrict utilization of the proposed steels to applications where component production and plant construction do not cause significant microstructural alterations, i.e. to components which are not welded. Furthermore implied complexity and additional cost might prohibit market entry or deeper market penetration. To overcome this drawback several heat treatment scenarios to increase/restore the tensile properties of rolled/recrystallization annealed or welded material, were developed.

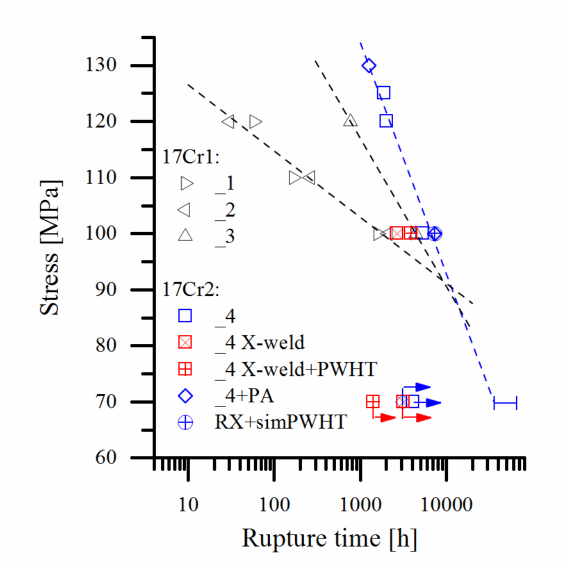
By heat treatment (cf. "RX+simPWHT" in Fig. 1a) almost doubled ambient temperature tensile strength values are possible in case of the 17Cr2 material. Alloying by a low amount of Ni in case of the 17Cr4 steel yields an increase in tensile strength of appr. 15 % (Fig. 1b: "17Cr4_4"), which is mainly caused by grain refinement. B addition (17Cr8) causes an increase of appr. 10 % (Fig. 1b: "17Cr8_4"). The reason for this is under investigation. In case of the 17Cr5 trial steel the W and Nb-contents were changed for increased tensile strength mainly by improved solid solution hardening (cf. Fig. 1b: "17Cr5_6") as long as no additional heat treatment is applied. The yield and tensile strength values achieved are approaching the range of AFM steels. Precipitation heat treatment for increased tensile strength of this alloying variant is still under investigation.

As a summary it can be stated, that simple heat treatment is mainly effective in increasing the ultimate tensile strength. If enhanced yield strength is desired, alloying and/or (thermo)mechanical processing is the measure of choice. Obviously there are several effective ways to adjust the short-term mechanical properties. The optimum way apparently depends on the designated application, which dictates detailed material specification.

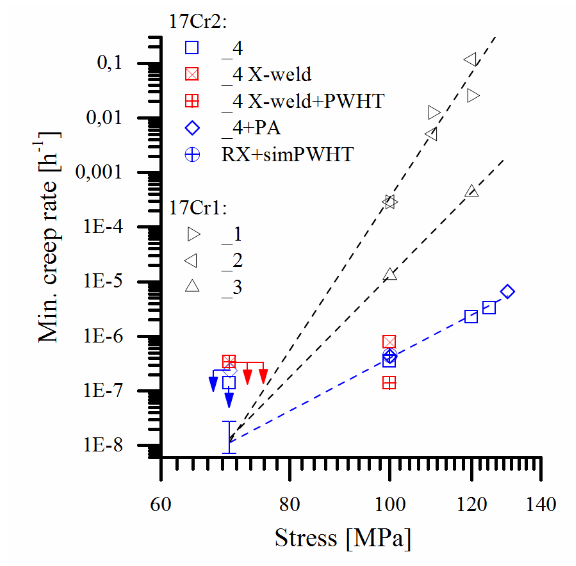
Base material creep properties

The overall creep testing duration of the various HiperFer grades to date cumulates to appr. 135.000 hours, from which a part is reported here. The 17Cr1 and 17Cr2 steels served as “working horses” to develop processing and heat treatment, to assess principal creep properties and models to describe them [13, 15]. In the beginning the materials were creep tested in the as-rolled condition (denoted by the _1 to _4-suffixes) to study the impact of varying thermomechanical processing. In the course of development several recrystallization (RX), heat treatment (precipitation annealing (PA) and (simulated) post-weld heat treatment ((sim)PWHT)) variants emerged to increase the creep performance of the base materials and welds.

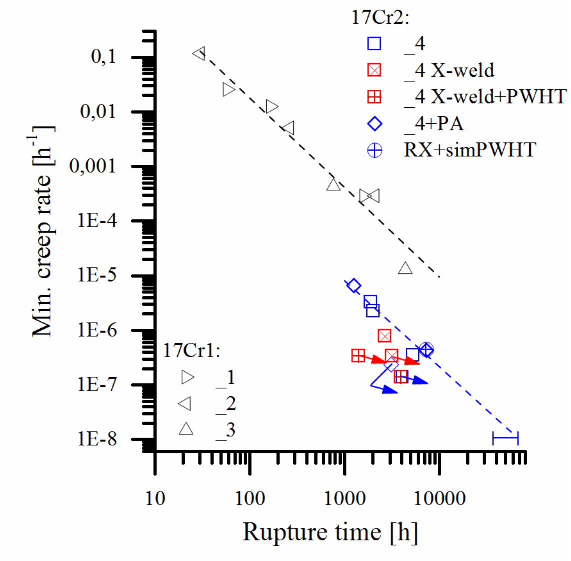
With changing processing schedule from parameter set _1 to _4 the creep life of the 17Cr1 and 17Cr2 steels increases (Fig. 2a), while the minimum creep rate drops (Fig. 2b). The rupture times and minimum creep rates of precipitation heat treated (“_4+PA”) and recrystallized and post-weld heat treatment simulated (“RX+simPWHT”) 17Cr2 material fit well into the data of the as-rolled steel (Fig. 2). Synopsis of the results obtained so far suggests, that the impact of thermomechanical processing fades out below a stress level of approximately 90 MPa (Fig. 2a) at 650 °C. Below this stress level / beyond this exposure time the thermomechanical treatment history is supposed to have no significant influence anymore with creep rupture mainly being controlled by particle stability.



a)



b)

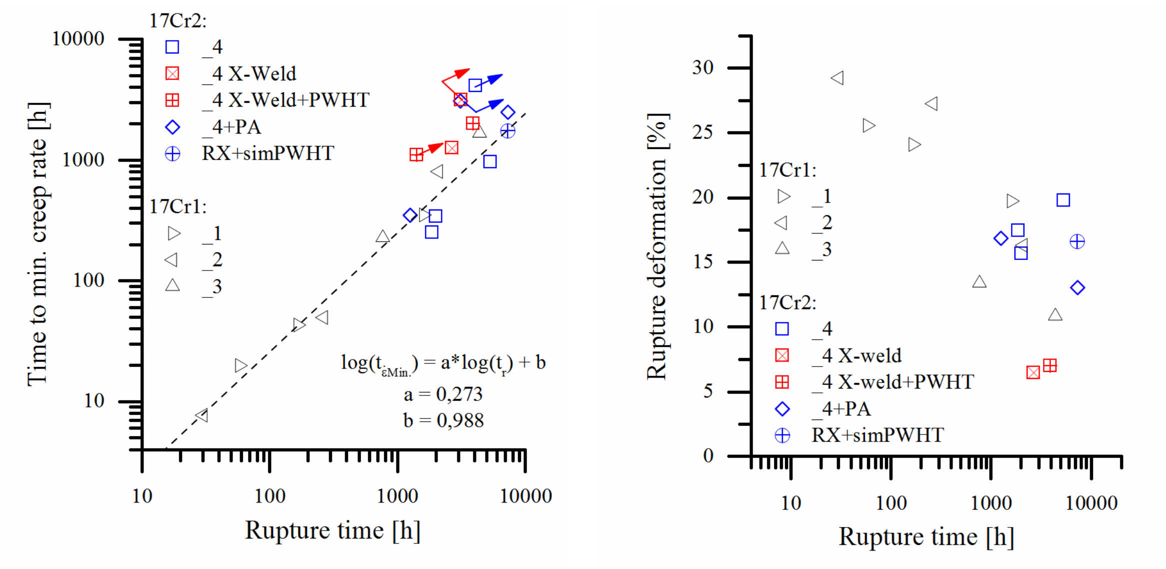


c)

Fig. 2: Results from 650 °C creep experiments: a) Stress vs. rupture time, b) minimum creep rate vs. stress and c) minimum creep rate vs. rupture time. Arrows indicate experiments in progress, which were excluded from fitting.

A stress level of 70 MPa was assessed for validation experiments from the proposed intersection of the batch data trend lines (range from 1 to $3*10^{-8}h^{-1}$ at appr. 70 MPa) in the given plot of minimum creep rate vs. stress (Fig. 2b). Corresponding long-term experiments are currently being executed at various 17Cr2 material variants (indicated by the arrows in Fig. 2a-c). Optimization of processing and heat treatment enabled a reduction of appr. two orders of magnitude in terms of minimum creep rate in case of the 17Cr2 variants (Fig. 2b and c). Based on the proposed range of minimum creep rates (Fig. 2b) the possible range of rupture times of the 70 MPa validation experiments was calculated to be within 35.000 to 75.000 hours and introduced into Fig. 2a and c (being fully aware that both the lower and upper values do disrespect a maximum allowable extrapolation factor of 3).

Ease of monitoring is a mandatory requirement for materials applied in power engineering. Related to creep a combination of sufficient duration of tertiary creep and rupture deformation is desired. All the 17Cr1 and 17Cr2 trial steels in all their processing and heat treatment states do well obey the same time to minimum creep rate / time to rupture relation (given in Fig. 3a). Considering the values of the regression constants (a = 0.273 and b = 0.988) it can be concluded, that more than 70 % of creep life lies within the short secondary and predominantly the tertiary stage of creep. Despite its creep strength HiperFer steel provides adequate creep rupture deformation (cf. Fig. 3b) if properly processed. The only exception from this was the thermomechanically over-sophisticated 17Cr1_3 batch, where severe processing resulted in comparatively coarse, heavily deformed grains [15], which limited ductility in tensile [12] and creep loading (Fig. 3b). Heat treatment after recrystallization is effective in restoring creep ductility and improving rupture time (cf. “RX+simPWHT” in Fig. 3b).

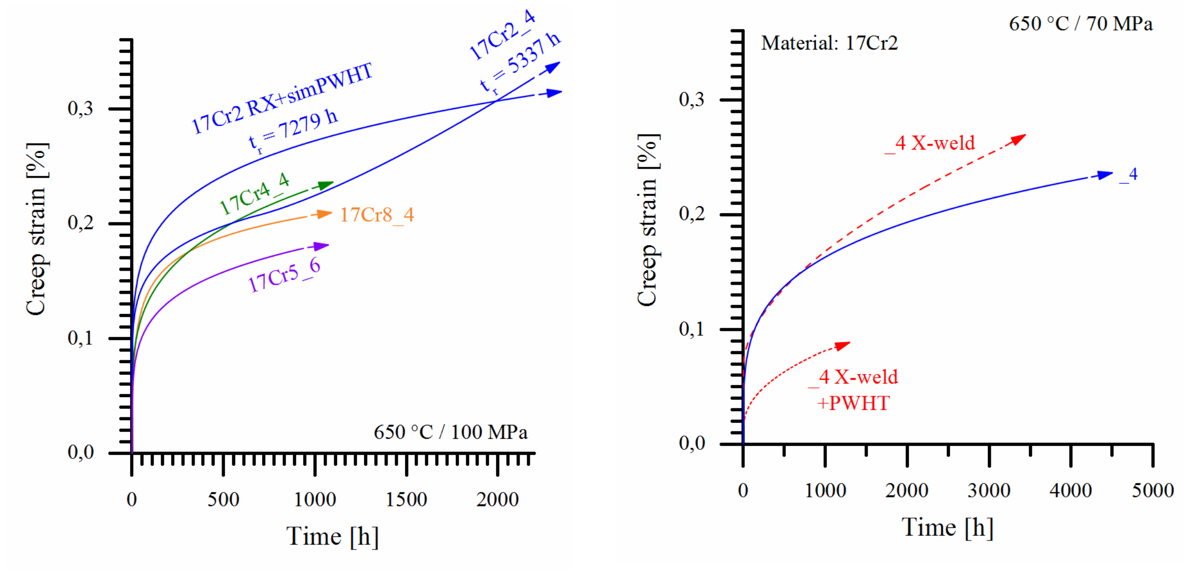


a) *b)*

Fig. 3: Results from 650 °C creep experiments: a) Minimum creep rate vs. rupture time and b) rupture deformation vs. rupture time. Arrows indicate experiments in progress, which were excluded from fitting.

Fig. 4 displays an overview of the 650 °C / 100 MPa creep curves: The as-rolled 17Cr2_4 steel failed after 5.337 hours (Fig. 4a), the recrystallized and post-weld heat treatment simulated variant (“17Cr 2 RX+simPWHT”) reached a rupture time of 7.279 hours. While the minimum creep rate remains merely unchanged (cf. Fig. 2b) the heat treatment performed after recrystallization shifts the time to minimum creep rate towards longer times and increases rupture ductility (Fig. 3b).

The experiments carried out at the as-rolled 17Cr4_4, 17Cr5_6 and 17Cr8_4 batches are still in progress and yield promising results so far (~ 1.100 h of creep). While in case of the 17Cr2_4 base composition the minimum creep rate and transition to tertiary creep appeared after ~ 960 h all the improved trial steels are still in the transient stage of creep with almost similar (17Cr4_4) or even lower accumulated creep strain (17Cr5_6 and 17Cr8_4). Under the assumption of similar time to min. creep rate / time to rupture relationships (Fig. 3a) the performance can already be estimated to be at least as good as, most probably even better than that of 17Cr2_4.



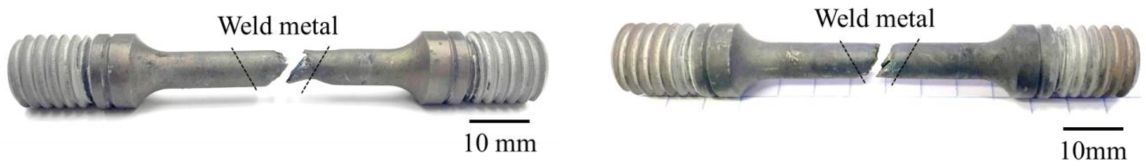
a) *b)*

Fig. 4: Exemplary creep curves of (a) various trial alloys from 650 °C / 100 MPa experiments and (b) from 650 °C / 70 MPa experiments carried out at rolled 17Cr2_4 and its cross-welds

The current result from the running low stress (70 MPa) base metal validation creep experiment is depicted in Fig. 4b together with two curves from cross-weld creep specimens. Like indicated in Fig. 2 and 3 all the low stress / long term experiments are still in the transient stage. The progress of all these experiments is not yet sufficient to draw final conclusions.

Cross weld creep properties

The as-welded / post-weld heat treated 100 MPa cross-weld creep specimens reached rupture times of 2.675 / 3.923 hours (Fig. 2a). Post-weld heat treatment thus increased the rupture time by appr. 30 %. The minimum creep rate / stress (Fig. 2b), minimum creep rate / rupture time (Fig. 2c) and time to minimum creep rate / rupture time Fig. 3a) relations of the cross-welds fall a little short of, but are still in reasonable agreement with, those of the base material.



a) *b)*

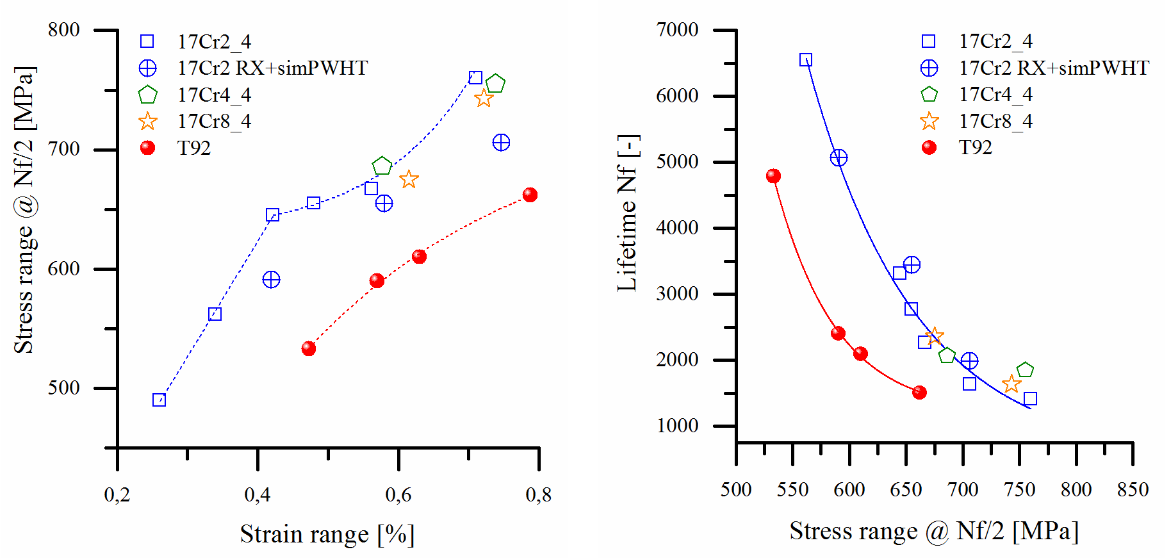
Fig. 5: (a) 17Cr2_4 X-weld and (b) 17Cr2_4 X-weld+PWHT creep specimen after rupture (650 °C / 100 MPa)

Creep deformation of the as-welded joint was mostly located in the heat-affected zone (Fig. 5a), while the post-weld heat treated specimen deformed almost uniformly (Fig. 5b). Both specimens ruptured within the weld metal with comparable overall rupture deformation (as-welded / +PWHT: 6.8 / 7 %), demonstrating that Type IV heat affected zone cracking is not an issue in HiperFer steels. The minimum creep rate of the post-weld heat treated specimen was the lowest measured so far (cf. Fig. 2b), even lower than that of the base metal. It can be concluded that post-weld heat treatment was effective in counterbalancing reduced dislocation strengthening of the heat affected zone by precipitation strengthening and improvement of weld metal ductility. In comparison to the base metal the cross-weld nevertheless ruptured prematurely, because of limited ductility of the weld metal (Fig. 3b). In contrast the as-welded specimen exhibited higher minimum creep rate than the base metal, deformation concentrated within the heat affected zone (Fig. 5a), but rupture was still located in the weld metal (Fig. 5a). For these reasons it is considered that the increase in minimum creep rate (cf. Fig. 2b) was caused by reduced dislocation strengthening of the heat affected zone, but failure by low ductility of the weld metal (like in case of the thermomechanically over-sophisticated 17Cr1_3 base metal, cf. Fig. 3b).

The progress of the 70 MPa cross-weld creep experiments (Fig. 4b and c) does not allow conclusions yet. Having in mind that the influence of processing history fades out in the long term it might - at least in case of the post-weld heat treated specimen - be speculated that the performance of the as-rolled base metal might be within reach.

Resistance to thermomechanical fatigue

Like reported in [8] ferritic high strength steels do provide favorable resistance to thermomechanical fatigue. In Fig. 6 the TMF testing results of the as-rolled, recrystallized and post-weld heat treatment simulated ("RX+simPWHT") 17Cr2 steel are plotted in comparison to commercial T/P92 material. The as-rolled HiperFer grade outplays the AFM material by far. Indicating superior strength potential its half-life stress range is almost 15 – 25 % higher than in case of T/P92 (Fig. 6a) at comparable strain ranges, what results in appr. doubled TMF lifetime (Fig. 6b). The cyclic stress vs. strain curves (Fig. 6a) exhibit contrarious behavior of the two material classes: While HiperFer hardens progressively because of dynamic precipitation of strengthening Laves phase particles on deformation induced dislocations the AFM steel softens because of pronounced recovery and (partial) recrystallization of the martensite lath structure [11] with increasing strain range. Consequently recrystallization and simulated post-weld heat treatment, which provokes precipitation, prior to TMF testing reduces the dynamic hardening potential of HiperFer (cf. "17Cr 2 RX+simPWHT" in Fig. 6a). Nevertheless the measured lifetimes fall close to the 17Cr2_4 main line (Fig. 6b) and remain superior to T/P92. At high stress/strain ranges the Ni-alloyed 17Cr4_4 and the B-added 17Cr8_4 steels exhibit similar cyclic hardening and lifetime like 17Cr2_4. The lower stress/strain range experiments are still in progress.

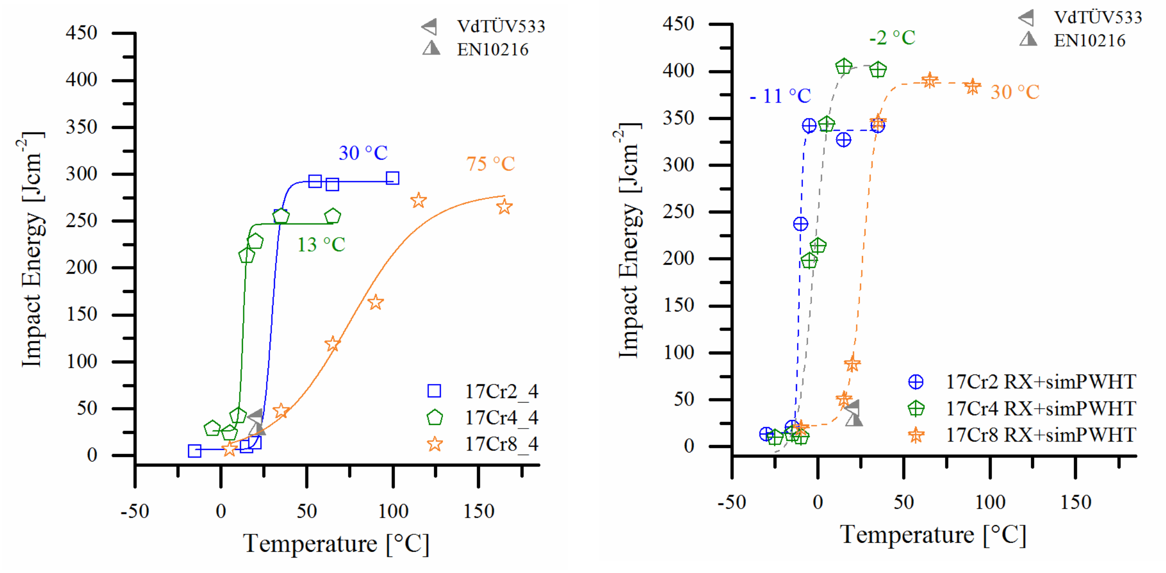


a) *b)*

Fig. 6: Results from out-of-phase thermomechanical fatigue experiments (50 - 650 °C, dT/dt = 10 Ks^{-1}, no dwell times). Cyclic stress vs. strain curve (a) and lifetime vs. half-life stress range (b) of as-rolled 17Cr2_4, 17Cr4_4, 17Cr8_4, recrystallized and post-weld heat treatment simulated ("RX+simPWHT") 17Cr2 steels and commercial T/P92.

Impact strength

According to the technical rules EN 10216 and VdTÜV 533 minimum Charpy impact energy values of 27 / 41 Jcm^{-2} are mandatory at ambient temperature. The impact strength of ferritic stainless steels is generally considered to be problematic, but if proper chemical composition, processing and heat treatment is applied this is not the case for HiperFer steel. Fig. 7a displays the brittle to ductile transition curves of the as-rolled 17Cr2_4, 17Cr4_4 and 17Cr8_4 steels, Fig. 7b the recrystallized and post-weld heat treatment simulated variants. In the as-rolled state the 17Cr2_4 base alloy and the B-added version 17Cr8_4 slightly remain below the limits, the Ni-added 17Cr4_4 composition provides an impact energy of 248 Jcm^{-2} at ambient temperature. In the post-weld heat treatment simulated state all the three materials surpass both limits. The correlations between chemical composition, processing, heat treatment, resulting microstructure and in consequence impact toughness are quite complex. For detailed information [16] may be consulted. Impact testing of the 17Cr5 alloy is in progress.



a) *b)*

Fig. 7: Brittle to ductile transition curves of (a) as-rolled and (b) recrystallized and post-weld heat treatment simulated 17Cr2, 17Cr4 and 17Cr8 steels

SUMMARY, OUTLOOK AND CONCLUSION

In appropriate heat treatment condition the 17Cr2 trial steel combines promising creep and cross-weld creep, short-term tensile, as well as supreme thermomechanical fatigue strength and viable Charpy impact properties. Thermomechanical treatment is an option to tune specific mechanical properties. For these reasons HiperFer can be considered as a high potential candidate material for a broad range of tubing, piping, blading and bolting applications in highly flexible, future power technology equipment like conventional back-up power plants, concentrating solar power plants or “Power-2-X” conversion systems.

Additional Ni-alloying of batch 17Cr4 for further improved impact strength was successful. Further changes in chemical composition regarding enhanced creep strength based on increased W- and Nb-levels (17Cr5) or alloying by B (17Cr8) are under investigation. All the new trial steels yield positive results so far and long-term validation creep experiments including temperature variation are in progress.

Future work will concentrate on optimization of the welding consumable and the welding parameters for the 17Cr2 composition to increase rupture ductility and strength up to the base metal level. Compositionally matched welding consumables and procedures optimized for the newer compositions will shortly follow. A new action will be started on the production of pilot tubes from the 17Cr2 and 17Cr4 composition.

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