



VHCF behavior and defect tolerance of modified bainitic 100Cr6 with a high retained austenite content

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ABSTRACT

Since the fatigue life of high strength steels, e.g., roller bearing steel 100Cr6, depends on microstructural defects, an improvement of their defect tolerance can enhance the performance of components. To improve the defect tolerance, the retained austenite content can be increased. Thus, in this work the high cycle and very high cycle fatigue behavior of two modified laboratory variants of 100Cr6, which have an increased content (1.5 wt%) of Al and Si, respectively, were analyzed since these alloying elements lead to higher fractions of austenite. Moreover, an industrially available variant with 0.6 wt% Si was analyzed as a reference. All steels were investigated in the bainitic condition and had austenite contents of about 20 vol.%. However, the distribution and chemical composition of the austenite differed. The presented results demonstrate that the effect of retained austenite on defect tolerance is caused by i) the increased ductility and ii) the local deformation-induced phase transformation. To strengthen the effects of the austenitic phase, a finer distribution as well as a lower austenite stability are useful. Consequently, to evaluate the effect of retained austenite on the fatigue behavior the content, the distribution and the chemical composition of the austenitic phase must be considered.

1. Introduction

In many industrial applications, the roller bearing steel 100Cr6 (EN ISO 683-17: 1.3505; AISI: 52100), and modifications thereof, are used in martensitic conditions, containing up to 10 vol% retained austenite [1]. This limitation of the retained austenite fraction in application has two main reasons: Besides the reduction of the tensile strength, the austenitic phase can transform to α' -martensite due to thermal or mechanical loadings. Since this phase transformation results in a volume expansion [2–4], extensive austenite – α' -martensite transformations lead to dimensional changes and thus, are not desired [2,5–7].

For analyzing the fatigue behavior of this kind of high strength steels, it must be considered, that they exhibit a high sensitivity to microstructural defects, e.g., non-metallic inclusions or pores, which reduce the fatigue life and lead to a large scatter in service life. Hence, the respective components, e.g., roller bearings, are usually designed with high safety factors. Moreover, the requirements regarding the cleanliness and the maximum size of defects are relatively high, which lead to high material costs.

In this context, Yajima et al. [8] and Clausen et al. [9] showed that an

increased content of retained austenite and the associated local deformation-induced transformation of austenite into α' -martensite leads to a significant increase of the defect tolerance of a material by suppressing the initiation and propagation of fatigue cracks at the defects. This is further confirmed by the work of Grigorescu et al. [10] and our own work [11], in which it was shown that locally restricted phase transformations in the vicinity of the fatigue crack initiation site lead to an improved fatigue life.

In summary, local phase transformations in the vicinity of crack-initiating defects are desired, while an extensive phase transformation in the whole material volume should be avoided to exclude dimensional changes. For this reason, the distribution of the retained austenite and its stability against deformation-induced transformation are crucial to exploit the advantages and to avoid the disadvantages risen above [12,13]. The austenite stability depends, among other factors, on the chemical composition of the austenitic phase [14,15].

Consequently, the content of retained austenite should be high enough to increase the defect tolerance of the 100Cr6, but a sufficient phase stability is required to avoid critical dimensional changes of the components. Sourmail et al. [16] have shown that Si increases the thermal and mechanical stability of the retained austenite in 100Cr6,

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Nomenclature			
AT	ambient temperature	E	tensile stress
BCC	body centered cubic	f	Young's modulus
DIC	digital image correlation	f_{eff}	frequency
EBSD	electron backscatter diffraction	HV	effective frequency
exp.	experimental	k	Vickers hardness
FCC	face centered cubic	N_f	correlation factor between s_a and σ_a
FEM	finite element method	R	number of cycles to failure
Fe_3C	cementite / carbide	R_m	load ratio
HCF	high cycle fatigue	$R_{p0.2}$	tensile strength
LOM	light optical micrograph	s_a	0.2 % yield strength
reg.	regression	T	displacement amplitude
SE	secondary electron	t_{pause}	temperature in fatigue and tensile tests
SEM	scanning electron microscope	t_{pulse}	time of oscillation pause in ultrasonic fatigue tests
VHCF	very high cycle fatigue	λ	time of oscillation pulse in ultrasonic fatigue tests
XRD	X-ray diffraction	σ	wavelength
A	elongation at fracture	σ_a	stress
area	Projected area of the defect perpendicular to maximum	σ_w	stress amplitude
		σ_a / σ_w	fatigue strength estimated by $\sqrt{\text{area}}$ approach
			representative of the relative stress intensity

since the addition of Si suppresses the formation of carbides during bainitization and thus, more carbon is available to stabilize the retained austenite [17]. Besides Si, also higher Al contents can increase the content of retained austenite and its stability. Al enhances the spheroidization of carbides, suppressing the formation of cementite networks [18]. Moreover, alloying with Al leads to the formation of nano-sized Al nitrides, which support the stability of fine grains during heat treatments, leading to an improved strength [19]. However, higher Al contents can also lead to a pronounced formation of oxide inclusions, which reduce the purity of the material [20,21], while Si contributes to a higher cleanliness [5].

On this foreground, in the presented work the fatigue behavior and defect tolerance of three modified 100Cr6 steels with an increased Si or Al content were analyzed. Therefore, fatigue tests in the high cycle (HCF), being already published for two steels in [22], and in the very high cycle (VHCF) fatigue regime were performed, using servo-hydraulic and ultrasonic fatigue testing devices. To analyze the influence of the deformation-induced phase transformation, the fatigue tests were performed at ambient temperature (AT) and at an elevated temperature of $T = 100^\circ\text{C}$, since higher temperatures suppress the transformation of austenite into α' -martensite [23,24]. For the evaluation of the defect tolerance, the fracture surfaces resulting from the fatigue tests were analyzed and the $\sqrt{\text{area}}$ approach established by Murakami [25] was used. Note that the results, discussion and conclusions presented in the previous work [22] are helpful to understand the results discussed in this work.

2. Investigated materials

In this research work, one industrially purchased and two newly developed and laboratory melted 100Cr6 steels, which differ in their chemical composition, were investigated. As described in detail in [26], the two laboratory alloys were melted in a vacuum-induction furnace, followed by ingot casting and hot forging at IEHK Steel Institute of RWTH Aachen University. The purpose of the ingot casting and

subsequent hot forging (performed in the temperature range of 1050 to 1250 °C), was homogenization of the materials. For the forging process, the casted ingots were gradually heated up to 1250 °C. The forged bars had a length of approximately 900 mm and a quadratic cross-section of 80 mm x 80 mm.

The chemical compositions of the investigated steels were measured using spark optical emission spectroscopy and are given in Table 1. The two laboratory melted steels have an increased content of Al and Si, respectively, which is higher than the limits given in DIN EN ISO 683–17 [1]. According to the respective contents of 1.5 wt-%, the developed alloying concepts are further called 1.5Al-100Cr6 and 1.5Si-100Cr6, while the industrial steel has the official designation “100CrMnSi6-4” according to DIN EN ISO 683–17 [1]. Hence, the 100CrMnSi6-4 represents an industrial reference. As shown in previous work [26], the presence of Al and Si in 100Cr6 steel leads to an increased retained austenite content and to a substantial influence on the stability of the austenitic phase.

Prior to the heat treatment of the materials at the Leibniz Institute IWT in Bremen, near-net-shaped blanks of the fatigue specimens were extracted from the forged bars of the 1.5Si-100Cr6 and 1.5Al-100Cr6 as well as from the industrially produced 100CrMnSi6-4 bars. Subsequently, the blanks were austenitized and then quenched in a salt bath to the bainitization temperature, which was held for 8 h for each condition. Due to the differences in the chemical composition of the three steels, variations in the retained austenite content would occur if the identical heat treatment parameters were used. As the aim of this research project was to analyze the effect of retained austenite on the fatigue behavior of bainitic 100Cr6, parameter analyses at dilatometer specimens were performed to find heat treatment parameters that lead to retained austenite contents being similar and between 15 and 30 vol% for all steels. Besides the retained austenite contents, also the mechanical properties were evaluated for the differently heat-treated dilatometer specimens. For this, instrumented cyclic indentation tests were performed, enabling the analysis of the hardness and the cyclic hardening potential [27]. Although the austenite content was the main criterium

Table 1

Chemical composition of the investigated steels in wt. %.

Steel	C	Si	Mn	Cr	P	S	Al	Ni	N
1.5Si-100Cr6	0.970	1.470	0.431	1.480	0.003	0.001	0.036	0.196	0.003
100CrMnSi6-4	0.930	0.590	1.090	1.460	0.010	0.010	<0.001	0.030	<0.007
1.5Al-100Cr6	0.970	0.300	0.421	1.480	0.003	0.002	1.440	0.194	0.003

for choosing the heat treatment parameters, also the hardness and cyclic hardening potential were considered, whereat the aim was to achieve high values of both, and especially the cyclic hardening potential. For each alloy 20 different combinations of austenitization temperature, austenitization time, bainitization temperature and bainitization time were considered. The heat treatment parameters chosen from this parameter study are shown in Table 2, respectively. The approach for the parameter study is described in detail in [27] for 100CrMnSi6-4 and 1.5Si-100Cr6, and was conducted analogously for the 1.5Al-100Cr6.

The heat treatments resulted in bainitic microstructures for all investigated steels, as shown in Fig. 1. The light optical micrographs reveal the finest bainite structure for the 1.5Si-100Cr6, while the 1.5Al-100Cr6 shows the coarsest one. This observation was further confirmed with electron backscatter diffraction (EBSD) phase maps. In the EBSD phase maps, the alloys with 0.6 and 1.5 wt% Si show more carbides and a finer distribution of the retained austenite, whereas the 1.5Al-100Cr6 exhibits large, blocky-shaped areas of retained austenite.

From the EBSD measurements significant differences in the retained austenite fractions might be concluded. However, note that the area and volume analyzed in EBSD is relatively small and thus, the deviations indicated by EBSD might be caused by local variations in the phase contents. Consequently, X-ray diffraction (XRD) analyses were additionally conducted, which enables the investigation of a bigger, and thus, a more representative material volume. The XRD measurements revealed nearly identical retained austenite contents for the different steels, i.e., 24 vol% for the 100CrMnSi6-4, 21 vol% for the 1.5Si-100Cr6 and 22 vol% for the 1.5Al-100Cr6.

The microstructures described above resulted in different monotonic properties, which were determined in tensile tests and are listed in Table 3. To evaluate the influence of the deformation-induced phase transformation, the tensile tests were performed at AT and at $T = 100\text{ }^{\circ}\text{C}$, as the higher temperature suppresses the phase transformation. Note that the results obtained for the laboratory melted steels are already shown in previous work [22].

At both temperatures, the 100CrMnSi6-4 reveals the highest Young's modulus E and tensile strength R_m , whereas the 0.2 % yield strength $R_{p0.2}$ of this steel is significantly smaller. Comparing the laboratory melted steels, no pronounced differences in the monotonic properties are seen. Only E is slightly smaller for 1.5Al-100Cr6 in relation to 1.5Si-100Cr6. Note that the elongations at fracture show small differences, which, however, can be neglected, when comparing the different variants.

Considering the influence of the increased temperature, nearly no influence is observed for the laboratory melted steels, which show only slightly higher $R_{p0.2}$ and smaller E at $T = 100\text{ }^{\circ}\text{C}$. Although the higher $R_{p0.2}$ at a higher temperature are surprising, it must be considered that the differences observed are relatively small and only a small number of specimens were tested. Moreover, for the temperatures investigated different control modes and measurement techniques were used, which can also influence the values determined. In contrast to that, the increase of $R_{p0.2}$ at $T = 100\text{ }^{\circ}\text{C}$ observed for 100CrMnSi6-4 is more pronounced and seems to have microstructural reasons, which must be considered by evaluating the fatigue results.

Table 2

Heat treatment parameters of the investigated steels.

Steel	Austenitization		Bainitization	
	Temperature in $^{\circ}\text{C}$	Time in min	Temperature in $^{\circ}\text{C}$	Time in h
1.5Si-100Cr6	900	60	240	8
100CrMnSi6-4	880	30	220	8
1.5Al-100Cr6	900	30	240	8

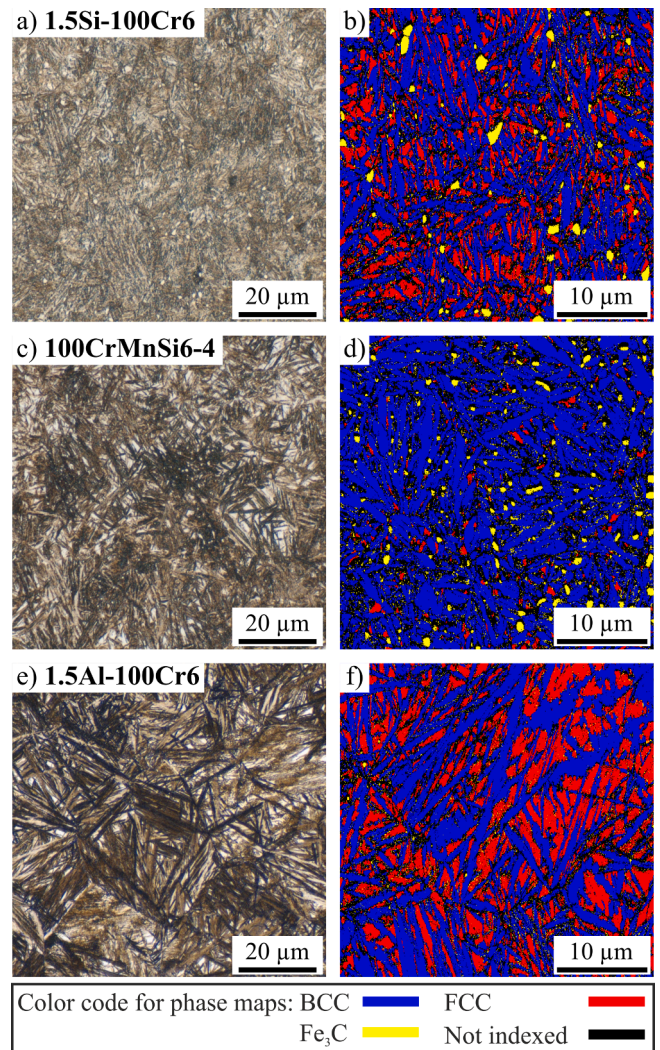


Fig. 1. Microstructure of the investigated steels shown as a), c), e) light optical micrographs (LOM) and b), d), f) EBSD (Electron Backscatter Diffraction) phase maps.

Table 3

Monotonic properties of the investigated steels obtained in tensile tests at ambient temperature (AT) and $T = 100\text{ }^{\circ}\text{C}$.

Material (test temperature)	R_m in MPa	$R_{p0.2}$ in MPa	A in %	E in GPa
1.5Si-100Cr6 (AT)	$2,174 \pm 7$	$1,749 \pm 12$	6.7 ± 0.7	207 ± 2
1.5Si-100Cr6 ($100\text{ }^{\circ}\text{C}$)	$2,197 \pm 6$	$1,808 \pm 14$	4.5 ± 0.6	188 ± 2
100CrMnSi6-4 (AT)	$2,357 \pm 96$	$1,054 \pm 17$	4.2 ± 1.2	215 ± 1
100CrMnSi6-4 ($100\text{ }^{\circ}\text{C}$)	$2,385 \pm 18$	$1,435 \pm 8$	5.8 ± 0.6	193 ± 4
1.5Al-100Cr6 (AT)	$2,154 \pm 39$	$1,684 \pm 16$	4.7 ± 2.7	198 ± 2
1.5Al-100Cr6 ($100\text{ }^{\circ}\text{C}$)	$2,179 \pm 5$	$1,785 \pm 15$	7.3 ± 0.7	176 ± 1

3. Experimental methods

3.1. Hardness measurements

As a prerequisite for using the $\sqrt{\text{area}}$ approach, the Vickers hardness HV was measured for all materials with a ZHU 250 top device from Zwick/Roell GmbH & Co. KG. Besides measurements at AT, for each material HV was determined at a temperature of $100\text{ }^{\circ}\text{C}$. Therefore, samples were heated up to $100\text{ }^{\circ}\text{C}$ in a furnace and subsequently, the hardness was measured. Note that the hardness was measured immediately after removing from furnace, while the sample temperature was recorded by pyrometry, showing that the temperature did not fall below

95 °C.

3.2. Tensile tests

For a comprehensive characterization of the monotonic properties of the materials, tensile tests were conducted at AT and $T = 100$ °C. At least two tensile tests were performed for each combination of material and temperature. All tensile tests were performed in accordance with DIN EN ISO 6892 [28] at specimens with the geometry given in [26], having a diameter in the gauge length of 4 mm. The tensile tests were performed at a universal testing machine of type 112 from Test GmbH.

To apply the elevated testing temperature of 100 °C, a radiation furnace was used, which was controlled based on a thermocouple of type K (NiCr-Ni). Since the extensometer cannot be used at this temperature, the furnace was not completely closed, enabling the strain measurement via digital image correlation (DIC). For DIC, a system of Me-go GmbH (Me-go measurement system 5 M) was used, including a digital camera and the respective software. Note that the tests at AT were conducted in strain control using a strain gage-based extensometer, while for the tensile tests at 100 °C force control was used, since the DIC measurements could not be used for test control.

3.3. Fatigue tests

Uniaxial fatigue tests were performed at AT and $T = 100$ °C. In the high cycle fatigue (HCF) regime, the fatigue tests were conducted stress-controlled on a servo-hydraulic testing device Schenck PSA 40 kN, using a sinusoidal load function with a frequency of $f = 10$ Hz and a load ratio of $R = -1$.

In addition to the analysis of the HCF behavior, which was already discussed for 1.5Al-100Cr6 and 1.5Si-100Cr6 in [22], the behavior in the very high cycle fatigue (VHCF) regime was investigated. For this, an ultrasonic fatigue testing system with the operating principle as described in [29] was used. In this testing system, the electrical power generator supplies a sinusoidal signal of $f = 20$ kHz. Based on the piezoelectric effect [30,31], the converter transforms the electrical signal into a mechanical oscillation with the same frequency (see Fig. 2a). To realize a standing wave, which leads to a minimum displacement and thus, a maximum stress in the middle of the gauge length, the specimens' Eigenfrequency must be close to the operating

frequency of 20 kHz. Thus, via dynamic finite element method (FEM) simulation, the specimen geometries were designed in accordance with the first Eigenmode. As input parameters for the FEM simulation only the elastic materials properties, i.e., Young's modulus and the Poisson's ratio, which were determined in tensile tests, are used, since only negligible proportions of plastic deformation are assumed [32–34]. Due to the alloy- and temperature-dependent Young's modulus, specimens with different geometries (see Fig. 2b) were used at AT and 100 °C, respectively. Moreover, an ultrasonic booster horn with a transformation ratio of 1:2.5 was used to ensure the required stress amplitudes σ_a in the specimens' gauge section.

Using linear elastic FEM simulations conducted with ABAQUS, the relation between the displacement amplitude s_a and the maximum stress in the specimen gauge length was calculated. Based on s_a measured during the fatigue tests with a sample rate of 100 kHz by a 1-axis laser vibrometer of type VibroOne from Poytec, displacement-controlled fatigue tests with a load ratio of $R = -1$ were realized. To achieve a laser signal with a quality and stability sufficient for test control, an appropriate reflecting foil was fixed on the bottom of the specimens. The values for the different correlation factors k between σ_a and s_a were determined by using Eq. (1) and are shown for each combination of material and test temperature in Table 4. Note, that only the stress amplitude σ_a is used to describe the fatigue results, enabling a geometry-independent comparison between the investigated steels.

$$k = \frac{\sigma_a}{s_a} \quad (1)$$

For the fatigue tests at $T = 100$ °C, different heating strategies were used. While in the fatigue tests at $f = 10$ Hz a radiation furnace was used

Table 4

Correlation factors k between stress amplitude σ_a and displacement amplitude s_a .

material (test temperature)	k in MPa/ μ m
1.5Si-100Cr6 (AT)	30.57
1.5Si-100Cr6 (100 °C)	34.18
100CrMnSi6-4 (AT)	46.96
100CrMnSi6-4 (100 °C)	34.23
1.5Al-100Cr6 (AT)	31.28
1.5Al-100Cr6 (100 °C)	33.20

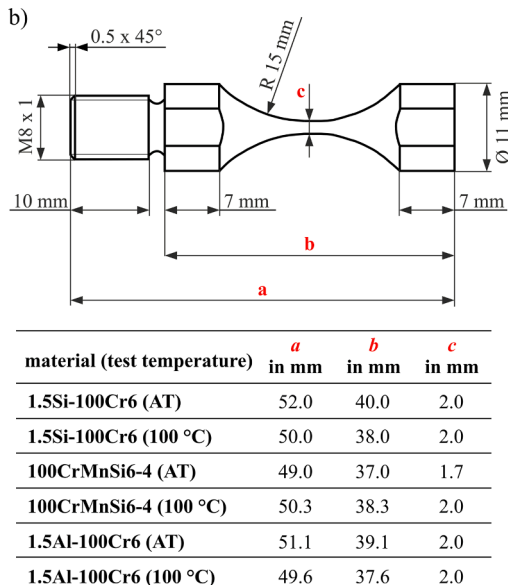
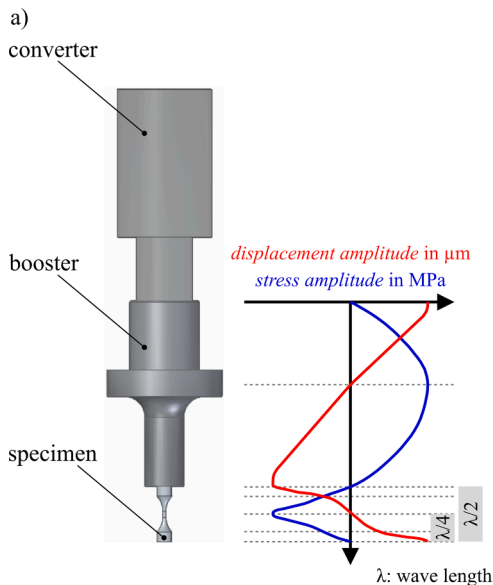


Fig. 2. A) schematical representation of the operating principle of the ultrasonic fatigue system and b) the geometry of the fatigue specimens tested at at and $T = 100$ °C.

to realize the elevated temperature, an induction heating system from TRUMPF Hüttinger, equipped with an induction coil and an induction generator with a nominal power of 10 kW, was used for the ultrasonic fatigue tests. In accordance with the tensile tests, the radiation furnace was controlled by means of a thermocouple of type K, whereas in ultrasonic fatigue tests a pyrometer of type Optris CTlaser LT fast (Optris GmbH, Berlin, Germany) was used to measure and control the temperature in the gauge length of the specimens.

To perform reliable fatigue tests with the ultrasonic fatigue testing system, a constant displacement amplitude, a constant pulse shape as well as short onset and decay times must be ensured throughout the whole test duration, which was achieved as shown in Fig. 3. Since continuous loading at ultrasonic frequencies results in a pronounced self-heating of the specimens, the fatigue tests were performed in pulse-pause-mode [35]. In this mode, short times of pulsing t_{pulse} are followed by a pause time t_{pause} for cooling. To increase t_{pulse} , the specimens were additionally cooled with compressed air.

The different materials have shown differently pronounced self-heating and hence, different pulse-pause ratios were applied to meet the maximum temperature limit of $T = 30^\circ\text{C}$, defined for the tests at AT. This resulted in different effective frequencies f_{eff} , which can be

determined by Eq. (2). While the laboratory melted alloys show only moderate self-heating, this effect was more pronounced for 100CrMnSi6-4. Thus, the effective frequency chosen for 100CrMnSi6-4 was $f_{\text{eff}} \approx 1,800$ Hz, while for 1.5Si-100Cr6 and 1.5Al-100Cr6 f_{eff} was about 8,900 Hz (see Fig. 3). For the fatigue tests at $T = 100^\circ\text{C}$, the pulse times were shortened to ensure a relatively constant test temperature close to 100°C . This leads to $f_{\text{eff}} \approx 1,250$ Hz for 100CrMnSi6-4 and $f_{\text{eff}} \approx 6,700$ Hz for 1.5Si-100Cr6, while for 1.5Al-100Cr6 $f_{\text{eff}} \approx 8,600$ Hz was reached, being close to the f_{eff} achieved at AT. Note that despite the differences in effective frequencies the actual test frequency is identical for all combinations of material and test temperature, i.e., 20 kHz.

$$f_{\text{eff}} = \frac{t_{\text{pulse}}}{t_{\text{pulse}} + t_{\text{pause}}} 20\text{kHz} \quad (2)$$

3.4. Application of the $\sqrt{\text{area}}$ concept

To thoroughly analyze the influence of the defects on the fatigue behavior, and hence the defect tolerance, the $\sqrt{\text{area}}$ approach established by Murakami [25] was utilized. Using Eq. (3), the theoretical fatigue strength σ_w can be calculated by considering the type of defect

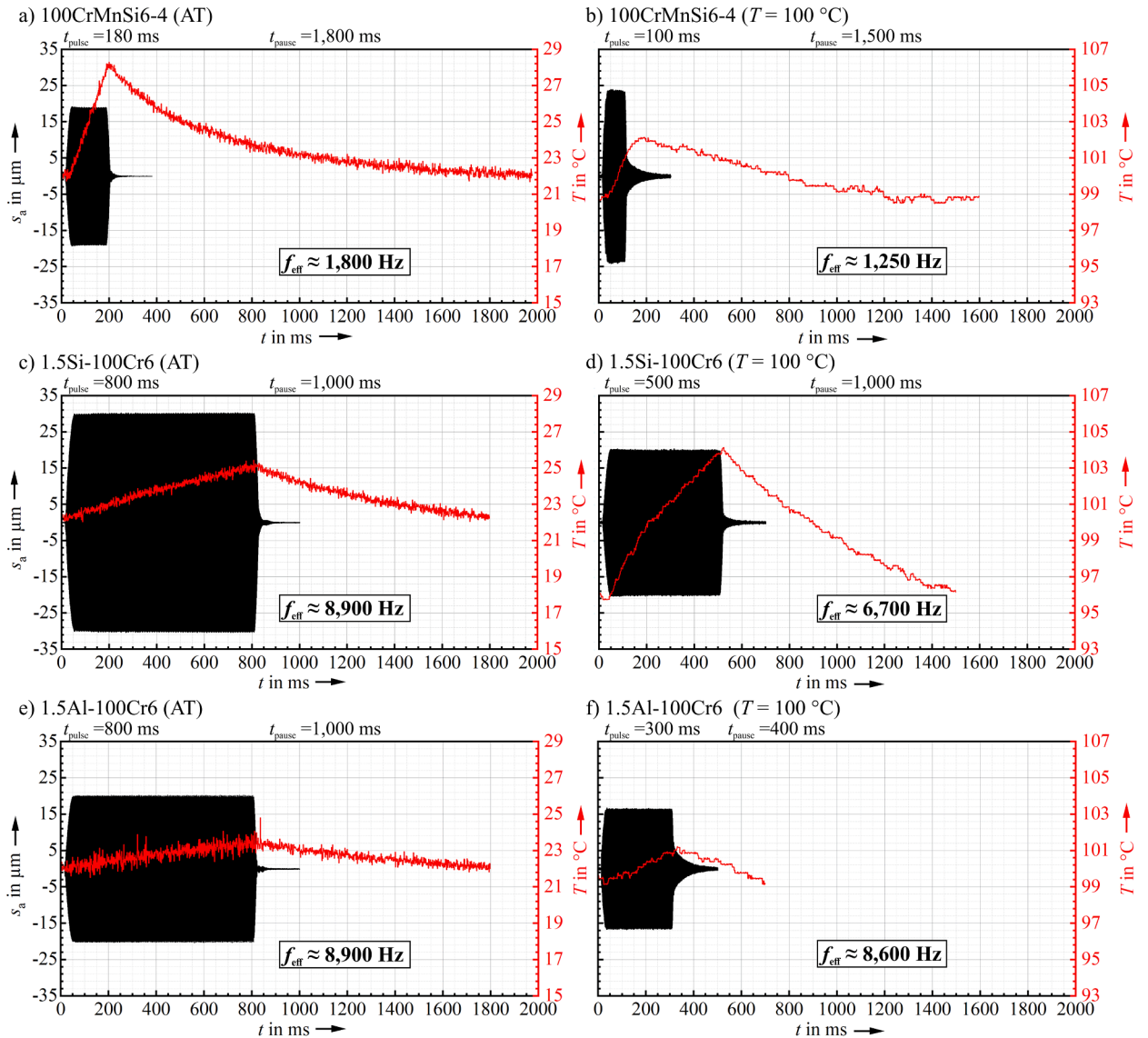


Fig. 3. Representative examples of the loading pulse shape and the resulting increase in temperature for the investigated materials obtained in ultrasonic fatigue tests performed at AT and at an elevated temperature of $T = 100^\circ\text{C}$.

and its size. Note that Eq. (3) with the factor 1.56 is only valid for volume defects, which were observed as crack initiation sites in all specimens investigated in this work. The defect size is considered by using the $\sqrt{\text{area}}$ value, being the square root of the projected area of the defect perpendicular to the maximum tensile loading. As a material parameter, only the Vickers hardness HV is utilized.

$$\sigma_w = \frac{1.56(HV + 120)}{(\sqrt{\text{area}})^{\frac{1}{6}}} [25] \quad (3)$$

Based on this approach, the defect tolerance was evaluated as shown in [36]. For this, the ratio of σ_a/σ_w was calculated for all specimens. This ratio represents the relative stress intensity at the crack-initiating defect and thus, is crucial for the fatigue life. Since higher relative stress intensities lead to earlier failure, σ_a/σ_w correlates well with N_f . Consequently, the σ_a/σ_w-N_f curves can be used to evaluate the fatigue behavior under consideration of the crack-initiating defects. Moreover, higher σ_a/σ_w at similar N_f indicate a higher defect tolerance of a material, since it can withstand higher relative stress intensities at microstructural defects for the same number of cycles [36]. Consequently, the defect tolerance can be evaluated by comparing the σ_a/σ_w-N_f curves.

3.5. Material characterization

Microstructure analyses were performed with a light optical microscope (LOM) Zeiss Axiophot. To visualize the phases, the samples were etched with 3 % nitric acid (Nital). For investigating the phase distribution, EBSD phase analyses were performed using a scanning electron microscope (SEM) Tescan Gaia 3 GMH. For the EBSD analyses, a detector EDAX Hikari operated at a working distance of 7 mm and a step size of 60 nm were used. To visualize the EBSD data, the software package OIM (Orientation Imaging Microscopy, version 8.0) from AMETEK (AMETEK GmbH, Meerbusch, Germany) was utilized. Since EBSD analyses require a high surface quality, the specimens were prepared according to the standard metallographic mechanical grinding and polishing procedures followed by electropolishing, which was performed at AT for 20 s at a voltage of 36 V in a commercial electrolyte consisting of 700 ml ethanol, 100 ml butyl glycol, and 78 ml 60 % perchloric acid.

In addition to the microstructural investigations, fracture surface analyses were performed using an FEI Quanta 600 FEG SEM device. For these analyses, a secondary electron (SE) detector, an acceleration voltage of 20 kV and a working distance of 10 mm were applied. In addition to that, the distribution of chemical elements in the vicinity of

the crack-initiating defects was determined by energy dispersive X-ray spectroscopy (EDS) using the software INCA. With the software Imagic, the sizes of the crack-initiating defects were determined in accordance with [25].

4. Results and discussion

4.1. Fatigue behavior in the HCF and VHCF regime at AT

The results of the fatigue experiments performed at AT are shown in Fig. 4a, revealing pronounced differences between the materials. In the HCF regime, the industrial reference shows a higher fatigue strength than the modified alloys, while the 1.5Al-100Cr6 shows the lowest HCF strength. In the VHCF regime, the 1.5Al-100Cr6 also reveals the smallest fatigue strength, but with a more pronounced difference to the other alloys than in the HCF regime. However, in the VHCF regime the laboratory melt 1.5Si-100Cr6 shows similar fatigue life as the industrial reference 100CrMnSi6-4. Notably, the material with the lowest 0.2 % yield strength shows the highest fatigue strength. Moreover, the laboratory melts show nearly identical tensile and 0.2 % yield strength, but significantly different fatigue strength in the HCF and VHCF regime.

When evaluating the fatigue behavior of high-strength steels, such as 100Cr6, the sizes of the crack-initiating defects must be considered since they mostly initiate HCF and VHCF failure. Consequently, fracture surface analyses were conducted, proving defect-based failure for all materials (see Fig. 5 and [22]). Note that in accordance with previous work on conventional fatigue specimens [22], the defects causing crack initiation in ultrasonic fatigue specimens of 1.5Si-100Cr6 are Al and Si oxides (see Fig. 5a). For the 1.5Al-100Cr6 one Al oxide and one hot crack were observed as crack initiation sites, which is also in accordance with [22]. However, in addition to this previous work, Al nitrides were the main type of crack-initiating defects in the VHCF regime (see Fig. 5c). This is assumed to be caused by the smaller volume of the ultrasonic fatigue specimens, leading to a lower probability of Al oxides in relation to conventional fatigue specimens investigated in [22]. Since the nitrides are smaller than the oxides they are expected to occur as crack initiation site in VHCF experiments due to the absence of the Al oxides. In contrast to the laboratory melts, the industrial reference 100CrMnSi6-4 reveals Al/Si nitrides as critical defect types in the HCF and VHCF regime.

For each specimen, the size of the crack-initiating defect was determined, which is visualized in Fig. 4b. For all variants it is obvious that the ultrasonic fatigue specimens have smaller crack-initiating defects

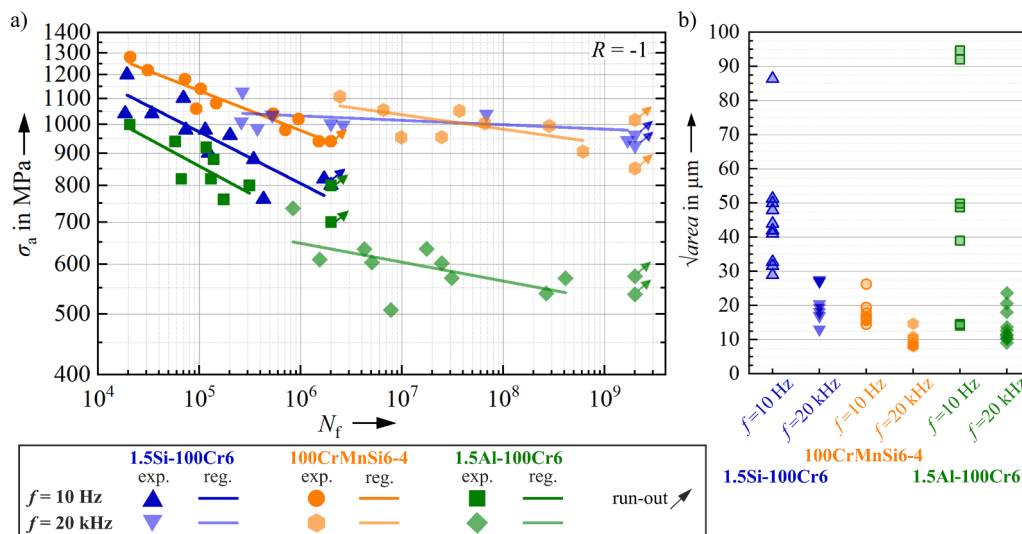


Fig. 4. A) $S-N_f$ curves and b) size distribution of the crack-initiating defects obtained after fatigue tests performed at AT for the investigated alloys.

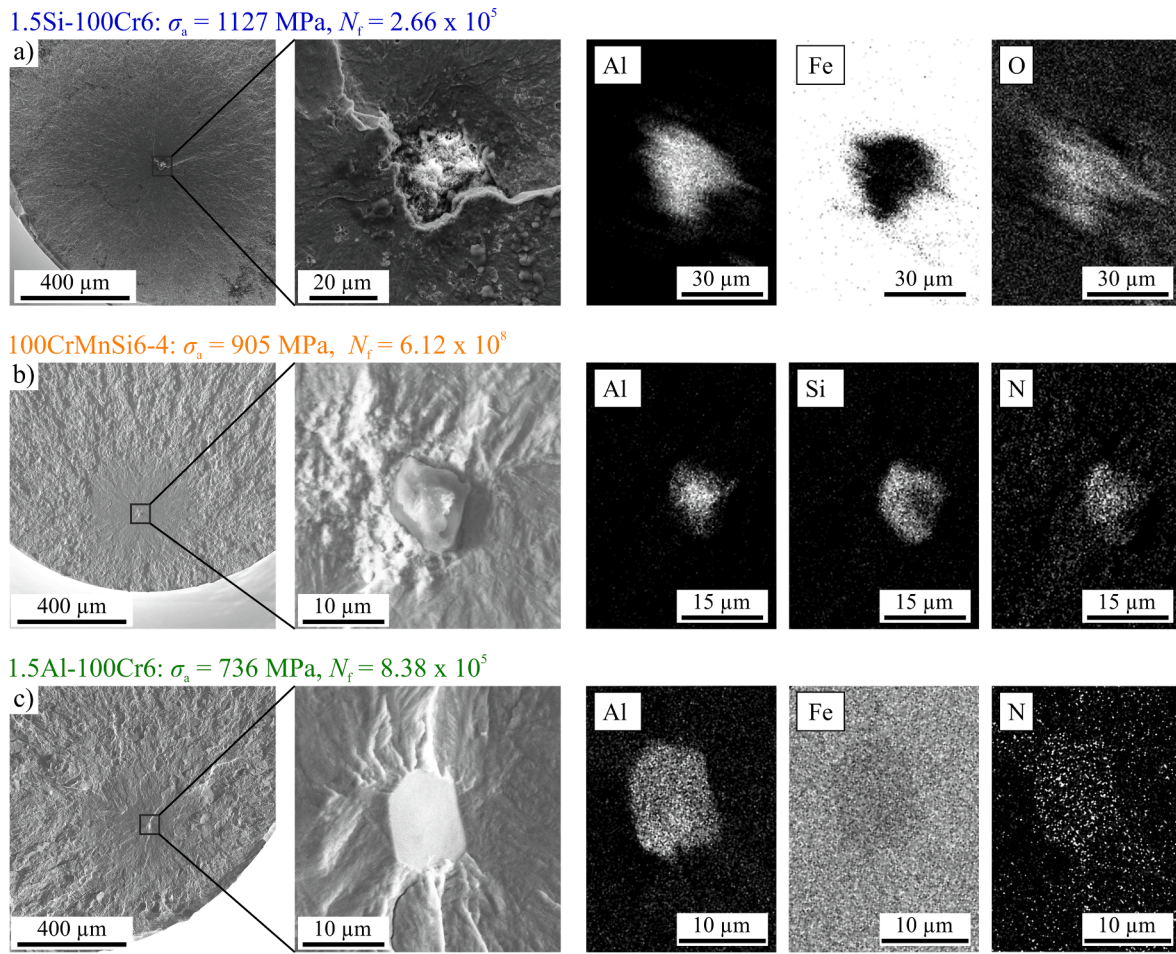


Fig. 5. Representative fracture surfaces obtained in ultrasonic fatigue tests performed at AT, showing typical crack-initiating defects as well as the chemical elements determined by EDS.

than the specimens loaded at 10 Hz, which is caused by the different volumes of the respective gauge lengths. Since the ultrasonic fatigue specimens are substantially smaller, the probability for the occurrence of bigger defects is lower. Comparing the sizes of crack-initiating defects between the different steels, the commercial 100CrMnSi6-4 has significantly smaller defects than the two laboratory melted steels, especially in the bigger specimens loaded at 10 Hz. Thus, for evaluating the fatigue behavior of the different materials, the defect-based failure was considered using the $\sqrt{\text{area}}$ -approach.

The results obtained via $\sqrt{\text{area}}$ approach are shown in Fig. 6, demonstrating nearly identical σ_a/σ_w-N_f curves for 1.5Si-100Cr6 and

100CrMnSi6-4 in the HCF regime. This indicates that the higher fatigue strength of 100CrMnSi6-4 observed in the HCF regime is mainly caused by the smaller defect sizes, overcompensating the lower 0.2 % yield strength. However, in the VHCF regime the 1.5Si-100Cr6 reveals even higher σ_a/σ_w , indicating an improved defect tolerance of this laboratory melt. Compared to the Si-alloyed steels, the 1.5Al-100Cr6 shows in the σ_a/σ_w-N_f curves substantially smaller fatigue life. Thus, the lower fatigue strength of the 100Cr6 alloyed with Al is not only caused by larger defects. Moreover, a lower defect tolerance in relation to the other materials can be assumed for 1.5Al-100Cr6, underlining the smaller fatigue strength of this alloy. This corresponds to the finer distribution of retained austenite observed in the Si-alloyed steels, leading to a higher probability of austenite presence in the vicinity of the crack initiation site. As discussed in [22], the relatively high plastic deformability of the austenitic phase as well as deformation-induced transformation of the austenite into α' -martensite suppresses crack initiation and propagation, leading to an improved defect tolerance.

Despite neglecting the defect type, the σ_a/σ_w-N_f curve obtained for 1.5Al-100Cr6 reveals only a small scatter in the VHCF regime. If the defect types observed, i.e., hot crack, Al oxides and Al nitrides, had a significant influence on the fatigue life, a higher scatter would occur. Since in 1.5Al-100Cr6 the influence of the defect type can be neglected, this aspect is also neglected for the comparison of the different alloys, since also oxides and nitrides were observed in the other alloys, respectively.

Furthermore, the results shown in Fig. 4a and Fig. 6 reveal a pronounced influence of the test frequency for the 1.5Si-100Cr6, while the other conditions do not show a significant influence of the test

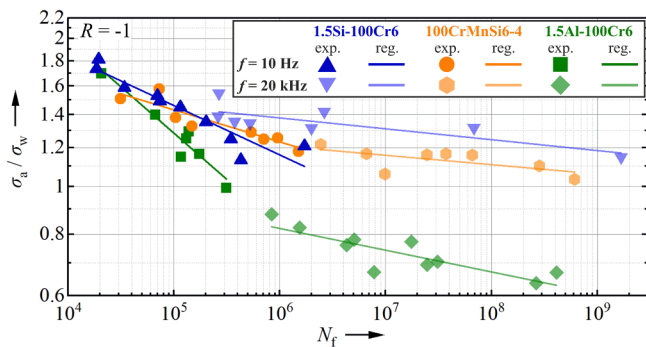


Fig. 6. σ_a/σ_w-N_f curves obtained at AT for the three investigated alloys in the HCF and VHCF regime.

frequency. Note that in Fig. 6 the smaller defect sizes in ultrasonic fatigue specimens are already considered and thus, the shift in σ_a/σ_w-N_f curves to higher values at a higher frequency cannot be explained with the different specimen geometry and the corresponding smaller defect sizes. Consequently, the strain rate significantly influences the fatigue behavior of 1.5Si-100Cr6. However, in conventional $S-N_f$ curves the influence of the frequency on the fatigue life of 1.5Si-100Cr6 is more pronounced, and thus, the effect of the bigger defects, which result from the bigger specimen volume, contributes to the differences observed in fatigue life between the tests performed at 10 Hz and 20 kHz.

4.2. Influence of an increased temperature ($T = 100^\circ\text{C}$) on the fatigue behavior

The results obtained at AT clearly indicate differences in the fatigue properties, which must be related to the microstructure. Since the different materials show deviations in the distribution of the retained austenite, the effect of deformation-induced austenite – α' -martensite transformation was analyzed in more detail by performing the same fatigue tests at an elevated temperature of $T = 100^\circ\text{C}$, which increases the austenite stability [23,24] and hence, suppresses the phase transformation. Thus, the comparison of the results obtained at AT and $T = 100^\circ\text{C}$ enables the analysis of the effect of deformation-induced transformation of the retained austenite. This comparison is depicted for each material in Fig. 7.

The results obtained for 1.5Si-100Cr6 show a reduction in fatigue strength due to the higher temperature in both, the HCF and VHCF regime (see Fig. 7a). This difference between the results obtained at AT and $T = 100^\circ\text{C}$ is even more pronounced in the VHCF regime. In contrast to the results obtained at AT, at $T = 100^\circ\text{C}$ no increase in fatigue strength due to a higher test frequency was observed, which correlates with the more pronounced temperature-induced decrease in fatigue strength.

Contrarily, the 100CrMnSi6-4 reveals no influence of the temperature on the fatigue behavior in the HCF regime, while for the ultrasonic fatigue tests a substantial reduction of the fatigue strength is observed at $T = 100^\circ\text{C}$ (see Fig. 7b).

In contrast to the other alloys, the 1.5Al-100Cr6 shows neither in the HCF, nor in the VHCF regime any influence of the elevated test temperature on the fatigue life (see Fig. 7c).

To ensure, that the interpretation of the effects of the increased temperature are not superimposed by differences in the sizes of the crack-initiating defects, also at the specimens tested at $T = 100^\circ\text{C}$ fracture surface analyses were performed. The results are illustrated in Fig. 8 and Fig. 9, showing similar sizes and, comparing Fig. 9 with Fig. 5, identical types of crack-initiating defects at both investigated

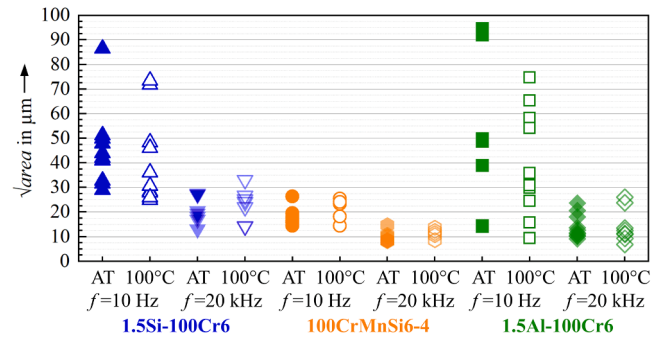


Fig. 8. Distribution of the defect sizes of the investigated steels obtained in HCF and VHCF fatigue tests performed at AT and $T = 100^\circ\text{C}$.

temperatures.

To thoroughly analyze the influence of the crack-initiating defects also for $T = 100^\circ\text{C}$, the $\sqrt{\text{area}}$ approach was applied. The σ_a/σ_w-N_f curves obtained at both investigated temperatures are compared in Fig. 10 for the different materials. These comparisons confirm the effects shown in $S-N_f$ curves: The 1.5Si-100Cr6 shows for both test frequencies a decrease in σ_a/σ_w due to the increased temperature, which is more pronounced for $f = 20\text{ kHz}$ (see Fig. 10a). From these results it can be assumed that i) the differences observed in the $S-N_f$ curves are not caused by differences in defect size and ii) that the defect tolerance of the 1.5Si-100Cr6 decreases at the higher temperature. For the commercial steel 100CrMnSi6-4, the results obtained in the $S-N_f$ curves are also confirmed and, in accordance with 1.5Si-100Cr6, a decrease in defect tolerance at the higher temperature is indicated in the VHCF regime (see Fig. 10b). Moreover, the 1.5Al-100Cr6 shows no influence of the increased temperature on the σ_a/σ_w-N_f curves, which also corresponds to the $S-N_f$ curves (see Fig. 10c).

Analyzing the results, it should be noted that the σ_a/σ_w-N_f curves partially end at values below 1, which is not in accordance to Murakami [25], as a stress amplitude smaller than the calculated fatigue strength resulted in fracture. However, since the calculation of σ_w is only based on the Vickers hardness HV , this deviation is assumed to be caused by an overestimation of the materials fatigue strength in the defect-free state, which is discussed in detail in previous work [37]. For this kind of steels, only using the hardness as material parameter for an estimation of the fatigue strength seems to be insufficient. However, the scatter in fatigue life observed in the σ_a/σ_w-N_f curves is relatively slow, and thus, it is assumed that the $\sqrt{\text{area}}$ approach can be applied and leads to qualitatively reasonable results.

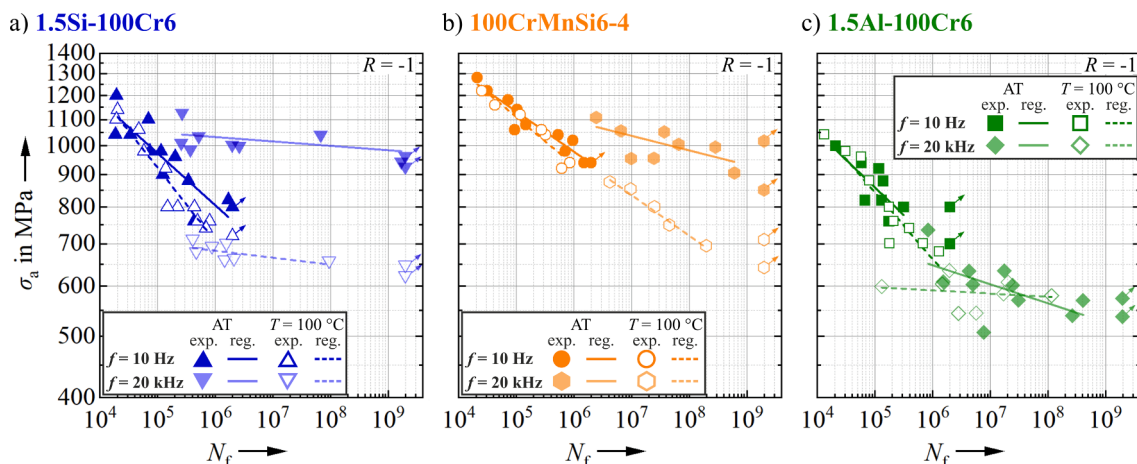
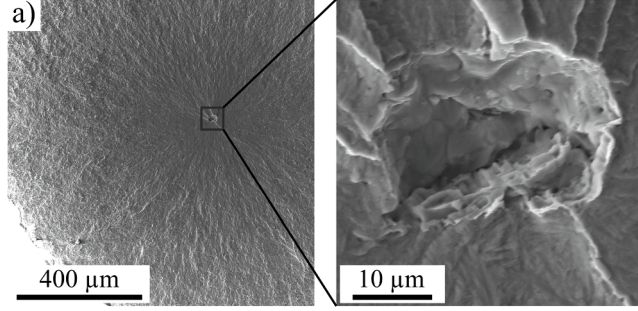
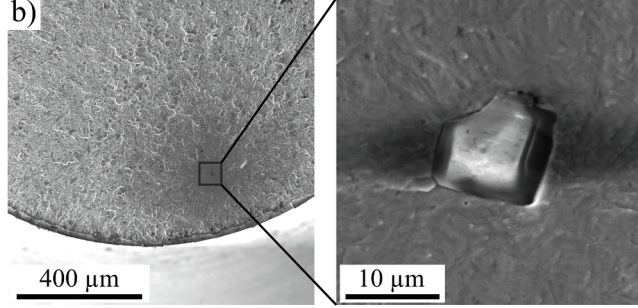


Fig. 7. Comparison between the $S-N_f$ curves obtained at AT and $T = 100^\circ\text{C}$ for the different materials.

1.5Si-100Cr6: $\sigma_a = 680$ MPa, $N_f = 4.69 \times 10^5$



100CrMnSi6-4: $\sigma_a = 876$ MPa, $N_f = 4.21 \times 10^6$



1.5Al-100Cr6: $\sigma_a = 583$ MPa, $N_f = 1.71 \times 10^7$

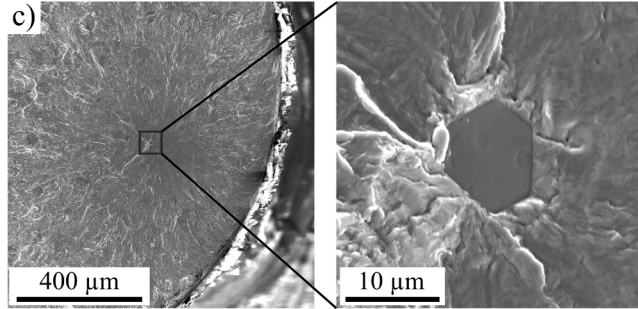


Fig. 9. Representative fracture surfaces obtained in ultrasonic fatigue tests performed at $T = 100$ °C for the different alloys.

4.3. Role of the retained austenite on the fatigue behavior and especially the defect tolerance

As shown in section 4.1, all materials show a relatively low scatter in fatigue life, which is, especially in the VHCF regime, not typical for this kind of high strength steels [38,39]. This low scatter can be mainly attributed to the high contents of retained austenite, having a relatively high plastic deformability and being distributed rather homogeneously in the material volume. For the 100CrMnSi6-4 it must further be considered that the distribution of the defect sizes is relatively narrow. However, also for this material condition the σ_a/σ_w-N_f curves indicate a high defect tolerance. Focusing on the VHCF regime, the 1.5Al-100Cr6 shows the smallest defect tolerance, which cannot be attributed to the content of retained austenite and must be caused by other microstructural aspects. As shown in Fig. 1 and previous work [22,26], the retained austenite regions in 1.5Al-100Cr6 are bigger and distributed less homogeneous in relation to the Si-alloyed variants. This leads to a lower probability of a presence of the austenitic phase in the vicinity of the crack initiation sites. Thus, the improvement of the defect tolerance due to the increased plastic deformability and the deformation-induced transformation of the austenitic phase is less pronounced in 1.5Al-100Cr6.

To discuss the influence of the deformation-induced transformation of the retained austenite into α' -martensite on the fatigue properties, the results obtained at AT and $T = 100$ °C are compared. As discussed in section 4.2, the different materials reveal different influences of the increased temperature on the fatigue life and thus, differently pronounced effects of phase transformation are assumed. In this context, it is expected that at $T = 100$ °C the deformation-induced phase transformation can be excluded. Since the 1.5Al-100Cr6 shows no effect of the increase in temperature, it is concluded that at both temperatures, AT and 100 °C, no phase transformation occurs. In contrast to that, both, the 1.5Si-100Cr6 and the 100CrMnSi6-4, show a decreased fatigue life and defect tolerance in the VHCF regime due to the higher temperature. Thus, for these materials a beneficial effect of the phase transformation can be expected at AT, since phase transformations in the vicinity of the defect lead to a deceleration of crack initiation and propagation [10], resulting in an increased defect tolerance and hence, fatigue life [11,40]. This beneficial effect is presumably suppressed at 100 °C, resulting in the decreased fatigue strength at $T = 100$ °C. This might explain the significantly smaller defect tolerance at AT of the 1.5Al-100Cr6 in the VHCF regime.

As already shown in [22], the 1.5Al-100Cr6 reveals a higher C content in the retained austenite than the 1.5Si-100Cr6, leading to a relatively high austenite stability, which can explain that no deformation-induced phase transformation is indicated for this material,

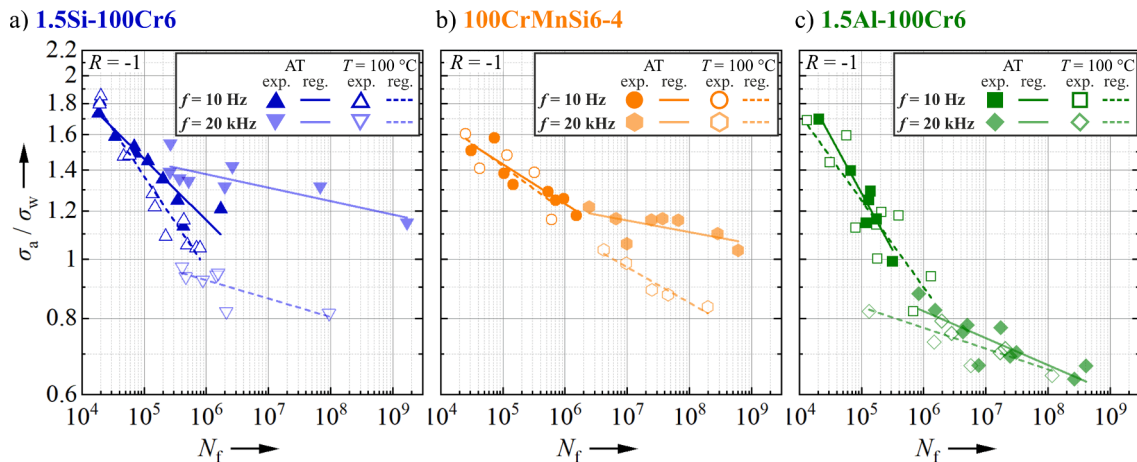


Fig. 10. Comparison of σ_a/σ_w-N_f curves obtained at AT and $T = 100$ °C for the steels investigated.

even at AT. Moreover, in [22] it was found that the distribution of the retained austenite is finer in 1.5Si-100Cr6 than in 1.5Al-100Cr6, which also supports the deformation-induced phase transformation, since the probability of retained austenite present at local stress concentrations, i. e., at defects, is higher. However, the XRD analyses in interrupted fatigue tests shown in [22] demonstrate, that no significant fatigue-induced change in phase distribution can be observed, which was also the case in the VHCF tests at all materials and the HCF tests on 100CrMnSi6-4. Moreover, measurements of the phase distribution of fractured tensile specimens did not reveal significant changes in the retained austenite content. Since XRD analyses only reveal information of the material volume, these measurements are not able to detect local phase transformations in the vicinity of defects. Thus, the effects of phase transformation indicated are assumed to occur only locally in the vicinity of crack initiation site, where increased stresses and hence, strains occur. Consequently, massive phase transformations, and thus, macroscopic dimensional changes were avoided for the materials investigated. Note that local analyses of the microstructure in the vicinity of the crack initiation sites were performed for 1.5Si-100Cr6 using Focused Beam Ion (FIB) technique. However, in these investigations the identification of the regions of austenite transformed into α' -martensite was not possible since the phase distribution before the cyclic loading is impossible to be analyzed retroactive. Thus, in future work it is planned to perform fatigue tests at specimens with artificial defects, which enables to observe the microstructural changes in the vicinity of the crack initiation site during cyclic loading.

Besides the effects described and explained above, for the 1.5Si-100Cr6 it becomes obvious that the temperature effect is more pronounced at 20 kHz than at 10 Hz. Note that a higher frequency should lead to a higher fatigue life, which can only be observed at AT, but not at $T = 100^\circ\text{C}$. In contrast to that, 1.5Al-100Cr6 shows no influence of the frequency at both temperatures. These observations can also be explained by the assumed differences in deformation-induced phase transformation. The work of Grigorescu et al. [10] demonstrate that higher strain rates result in more pronounced transformations of the austenite into α' -martensite. Thus, a more pronounced phase transformation can be assumed at 20 kHz than at 10 Hz. Since the 1.5Al-100Cr6 seems to show no phase transformation, no effect of the higher strain rate is overserved, while for 1.5Si-100Cr6 the effect of higher fatigue strength at 20 kHz corresponds to a more pronounced austenite – α' -martensite transformation. In correspondence to that, this effect of the strain rate is not seen at $T = 100^\circ\text{C}$, where the phase transformation can be excluded. Moreover, these interpretations are in accordance with the results obtained for 100CrMnSi6-4, showing only for the tests performed at 20 kHz an effect of the increased temperature. Consequently, for the 100CrMnSi6-4 it can be assumed that for 10 Hz no phase transformation occurs neither at AT nor at $T = 100^\circ\text{C}$, while at 20 kHz the effect of deformation-induced phase transformation occurs at AT. Based on the presented results, it can be concluded that the stability of the retained austenite in 100CrMnSi6-4 is smaller than in 1.5Al-100Cr6, but higher than the stability of the retained austenite present in 1.5Si-100Cr6. However, this hypothesis needs to be verified in future analyses, i.e., tracing the deformation-induced change in phase distribution in the vicinity of the crack initiation site in interrupted fatigue tests at specimens with artificially introduced defects.

In summary, the deformation-induced transformation of retained austenite into α' -martensite can be used to explain all the effects seen in the fatigue tests, even it was not measured in detail. Note that the verification of this assumption requires high-resolution microscopy combined with interrupted fatigue tests, which was not able to perform until now, but is planned for future work. However, the presented results indicate that an increased retained austenite content improves the defect tolerance of 100Cr6 and that local deformation-induced phase transformations in the vicinity of the crack-initiating defects might increase the defect tolerance significantly.

5. Summary and conclusion

In the presented work, the HCF and VHCF behavior of two modified variants of the steel 100Cr6, which contain an increased content of 1.5 wt% Al (1.5Al-100Cr6) and Si (1.5Si-100Cr6), respectively, were investigated and compared to a commercial alloy with 0.6 wt% Si (100CrMnSi6-4). Since preliminary work showed that higher contents of retained austenite increase the defect tolerance, the focus of the analyses was placed on the dependency of the defect tolerance on the retained austenite.

Hence, all alloys were bainitized to achieve similar retained austenite fractions of about 20 vol%. However, due to the different alloy compositions and the individual heat treatment parameters differences in the distribution and chemical composition of the austenitic phase were achieved between the alloys investigated. The fatigue tests and microstructural analyses performed led to the following conclusions:

- Homogenously distributed retained austenite significantly increases the defect tolerance of 100Cr6, since it increases the probability of the presence of retained austenite in the vicinity of crack-initiating defects.
- Local deformation-induced transformation of the retained austenite into α' -martensite is assumed to significantly improve the defect tolerance and thus, the fatigue strength. This effect seems to be more pronounced in ultrasonic fatigue tests since the phase transformation is enhanced at higher strain rates.
- Increasing the Si or the Al content of 100Cr6 leads to a higher defect tolerance, where Si-alloying revealed more promising results, since for this variant also the beneficial effect of the deformation-induced phase transformation comes into account, while the Al-alloyed variant indicated no phase transformation. This is assumed to be caused by differences in the austenite stability. However, this effect is superimposed by the differences in heat treatment parameters and must be analyzed more thoroughly in future work.
- For both alloy variants with increased Si content, the results indicate a deformation-induced phase transformation. However, this seems to occur only locally, and thus, excessive phase transformation was avoided. In the context of component design, this is a prerequisite to ensure a dimensional accuracy and thus, these alloy concepts are applicable.
- When evaluating the effect of retained austenite on the fatigue behavior, the fraction, the austenite stability, and the distribution of the austenitic phase must be considered.

CRedit authorship contribution statement

P. Ostermayer: Writing – review & editing, Writing – original draft, Visualization, Validation, Investigation, Data curation, Conceptualization. **B. Blinn:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration. **U. Krupp:** Writing – review & editing, Resources, Project administration, Conceptualization. **T. Beck:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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