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Deformation induced martensite formation in metastable austenitic steel during in situ fatigue loading in a scanning electron microscope

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Abstract. Aim of the study is to identify quantitatively the influence of deformation-induced phase transformation on the fatigue damage of a metastable austenitic steel during loading in the high cycle fatigue regime. Cyclic deformation tests were carried out in situ in a scanning electron microscope (SEM) in combination with automated electron backscatter diffraction (EBSD) used for phase analysis and crystallographic orientation mapping. The in situ experiments were supported by ex situ cycling in a servohydraulic testing machine. The examined metastable austenitic steel (AISI 304L) transforms diffusion less from the fcc austenite lattice to the bcc α′ martensite lattice either spontaneously at very low temperatures or at room temperature when a critical value of monotonic or accumulated cyclic plastic strain is exceeded. The experiments showed that already after some initial 10,000 cycles of fatigue loading at stress amplitudes close to the fatigue limit a nucleation of martensite occurs as needles near activated slip systems as a consequence of localized plastic deformation. Once first microstructurally short cracks have nucleated, strong martensitic transformation occurs within the plastic zone ahead of the crack tip. Due to the higher specific volume the martensite is considered to shield the crack tip, i.e., transformation-induced crack closure takes place. The role of deformation-induced phase transformation on (i) crack initiation and (ii) the mechanism of fatigue microcrack propagation is discussed in detail in the present paper.

1. Material and Experimental Details

The metastable austenitic stainless steel (AISI 304L) was delivered as rods with 25.5mm diameter. The chemical composition is given in Table 1. Before fabrication of specimens a solution annealing for 1h at 1050°C has been carried out to coarsen and homogenise the microstructure resulting in an average grain size of 75µm. After the heat treatment the microstructure was completely austenitic with a slight amount of linear arranged δ ferrite.

Table 1. Chemical composition of the austenitic stainless steel investigated

Shape	Alloy	Fe	C	Cr	Ni	Si	Mn	Cu	Mo
rod	AISI 304	Base	0.03	18.1	8.75	0.62	1.85	0.54	0.37

Cylindrical shallow notched specimens were used [1] to carry out the short crack growth experiments in a MTS servohydraulic testing system. In order to carry out in situ experiments in a Philips XL30 LaB_6 scanning electron microscope (SEM) a piezo driven testing system was newly developed for fatigue loading of flat specimen (Fig. 1.).
All tests were executed under load control at a load ration of R=-1 and a frequency of 5 Hz at room temperature. The chosen stress amplitude of $\Delta\sigma/2$ = 230 MPa was slightly above the fatigue limit (10^7).

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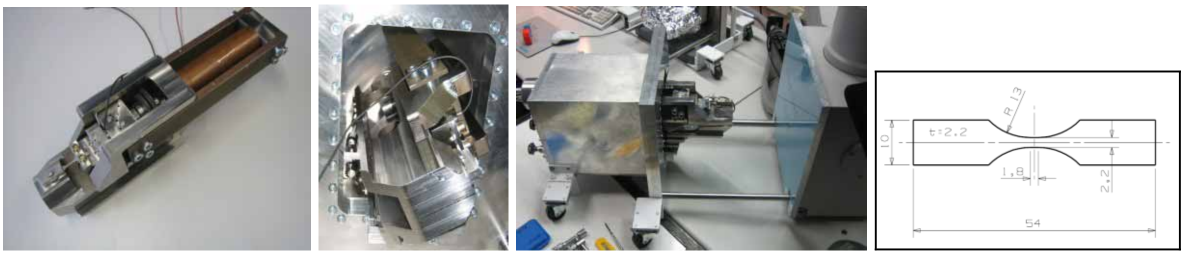


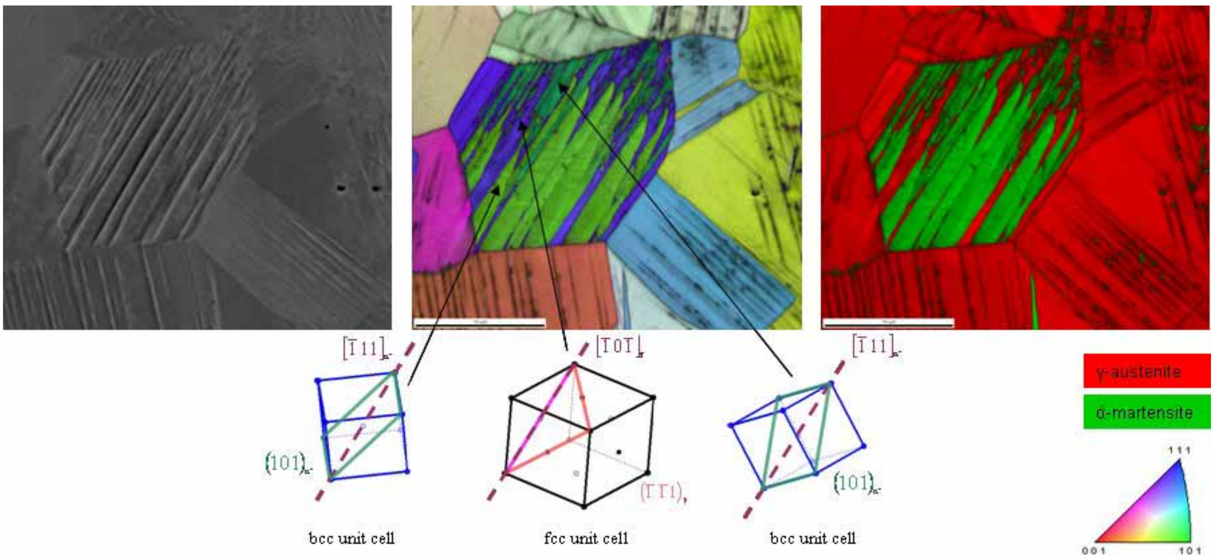
a) b) c) d)

Fig. 1. In situ fatigue testing system, a) piezo driven miniature testing system, b) 70° tilt for in situ electron backscatter diffraction (EBSD), c) SEM chamber enlargement, c) flat in situ specimen

The ex situ experiments were interrupted after a certain number of cycles for crack investigation in the SEM. An automated EBSD (TSL OIM^{TM}) was used for determination of the local lattice orientation and for an analysis of the phase transformation. It should be noted that smooth and deformation-free surfaces are necessary for OIM investigation. Therefore the specimens were electrochemically polished before testing.

2. Formation of martensite under cyclic loading in the HCF regime

During cyclic loading at the stated stress amplitude stated, a locally high plastic activity arrises. Nearly every grain shows slip markings. Close to activated slip systems of the highest Schmid factors (almost 0.5) a transformation of austenite to lamellae of martensite can already be detected after some initial 10.000 cycles even though a global threshold for transformation has not been reached [2] (Fig. 2.). From the EBSD phase data the α´ martensite can be determined as the body centred cubic (bcc) lattice structure. The existence of the hexagonal close packed (hcp) ε martensite could not be proven. Most likely, ε martensite can be considered as a transition phase in the transformation from the face centred cubic (fcc) austenite to α´ martensite [3]. Another reason for not detecting ε martensite could be the insufficient resolution of the SEM used for the very small volume fraction of this martensitic phase.



a) b) c)

Fig. 2. Nucleation of α´ martensite near activated slip systems according to the *Kurdjumov-Sachs* relationship, a) SEM micrograph, b) EBSD inverse pole figure map, c) EBSD phase map

The crystallographic relationship of austenite matrix and formed martensite was calculated from the EBSD orientation data. Within some scatter it was found that a close-packed lattice plane of the fcc austenite is parallel

to a close-packed lattice plane of the bcc martensite and within this parallel planes close-packed lattice directions are parallel.
This kind of crystallographic orientation relationship is described by the *Kurdjumov-Sachs* (K-S) orientation relationship [4] (Fig. 2., (1)).

$$(\bar{1}\,1\,\bar{1})_{\gamma} \parallel (101)_{\alpha'} \wedge [\bar{1}\,\bar{1}\,0]_{\gamma} \parallel [\bar{1}\,\bar{1}\,1]_{\alpha'} \qquad (1)$$

With 4 possible plains and 6 equivalent directions per plain this relation leads to 24 (12 twin related) variants of martensite. The two variants illustrated in Fig. 2. are formed on the same schema and are twin related.

3. Crack initiation and short crack growth

At the stress amplitude applied, active cracks can be observed very early in fatigue life although the density of cracks is very small. Only one or two cracks per sample side were found in the absence of any diffusionless phase transformation. In the majority of cases the initiation sides are located at twin boundaries (TB) as it is often observed in stable austenitic stainless steels [5,6].
The initial propagation takes place as shear-stress-controlled stage 1 crack propagation along the initiating twin boundary. In this stage no formation of α′ martensite takes place in connection with crack growth. After leaving the TB the cracks propagate transcrystalline perpendicular to the loading direction on crystallographic crack planes with low indices, such as {100} and {110} (Fig. 3.).

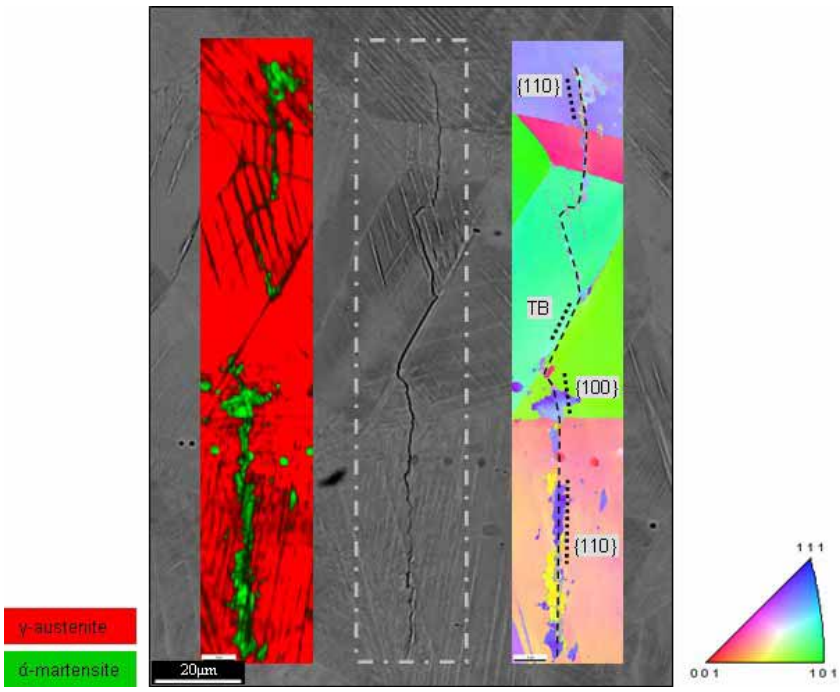
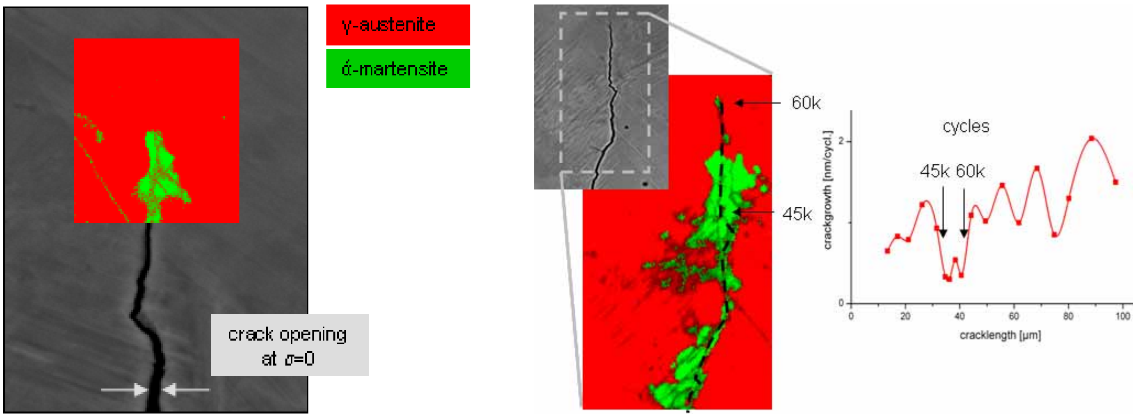


Fig. 3. Short crack in metastable stainless steel, left: EBSD phase map, right EBSD inverse pole figure map

This kind of crack propagation is described in ref. [1] as stage 1b or in refs. [5,7] as a *plastic blunting/resharpening mechanism* stage 2 crack growth where two adjacent {111} slip systems are involved.
As a consequence of crack propagation in the described manner, formation of α′ martensite in the plastic zone of the crack tips take place. Similar to the martensite lamellae described above, the crystallographic orientations obey the *Kurdjumov-Sachs* relation. The extend of the transformed area and the crystallographic orientation are fluctuating along the crack path.

4. Transformation induced short crack closure

Transformation induced crack closure of long cracks in metastable stainless steels is a well-known phenomenon [8,9]. The volume increase of 2-5% of the formed α´ martensite that nucleates in the plastic zone of long cracks leads to compression stresses in the wake of propagating cracks. This leads to a premature contact of the crack surfaces and hence to a reduction of the effective cyclic stress intensity factor.



a) **b)**

Fig. 4. Transformation induced crack closure, a) short crack is still open at zero loading, b) propagation through large transformed areas results in a strong decrease in crack growth velocity

In the case of short cracks there are also indications of transformation induced crack closure similar to long cracks. It was observed that the cracks are still open at zero load, when the zone of transformation at the crack tip is large (Fig. 4. a). Furthermore the crack is retarded while propagating through these large transformed areas (Fig. 4. b). As a subject of ongoing work an interferometric strain/displacement gauge has been applied for measuring local displacements in order to study the phenomenon of transformation induced short crack closure in more detail.

5. Summary

The influence of martensite formation on initiation and growth of short cracks in the high cycle fatigue regime was surveyed. The main results can be summarised as follow:

1. After some first 10.000 cycles of fatigue loading, a nucleation of α´ martensite takes place in local areas of the microstructure although the global threshold for transformation is not reached.

2. The α´ martensite forms needles near activated slip systems because of high local plastic deformation.

3. The correlation of the crystallographic orientation of the fcc austenite and the bcc martensite lattice follows the *Kurdjumov-Sachs* relationship.

4. Short cracks initiate mostly at twin boundaries in absence of any formerly developed α´ martensite.

5. After propagation along the initial twin boundary in stage 1 crack growth, the crack continues to grow perpendicular to the loading direction by a crack growth mechanism where two adjacent {111} slip systems are involved. This crack growth mechanism leads to crack planes with low indices such as {100} and {110}.

6. In the plastic zone of the growing short cracks a formation of α´ martensite occurs. Due to the volume expansion of 2-5% a crack closure effect seems to retard the short crack growth. By use of an interferometric strain/displacement gauge for measuring local displacements at very high resolution this phenomenon will be studied in more detail in the ongoing work.

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