Institut für Eisenhüttenkunde
RWTH AACHEN UNIVERSITY

Referee report

on the doctoral thesis by

Ing. Veronika Mazánova, submitted to the Faculty of Mechanical Engineering at Brno University of Technology, entitled:

"Short Crack Growth in Materials for High Temperature Applications"

1 Technical Background and Literature Review

The PhD project of Veronika Mazánova deals with a fundamental analysis of the fatigue and short crack growth behaviour in the high-temperature austenitic steel SANICRO25 (Sandvik trade name for 22Cr25NiWCoCu), that is designed for structural applications in coal-fired power plants due to its excellent creep and oxidation resistance.

The work follows extensive research work of Professor Polák´s group on cyclic plasticity and the formation of persistant slip bands (PSB), which manifest themselves by the occurrence of persistant slip markings (PSMs) at the surface of cyclically deformed metallic materials. Polák's model is carefully reviewed and acts as a baseline for the interpretation of the surface relief formation during fatigue of SANICRO25. Probably, the most well-known approach to explain the ladder-like nature of PSBs is the EGM model, named after Essmann, Gösele and Mughrabi. They assume dislocation dipole annihilation in the PSB walls that lead to the formation of point defects. In the case of vacancy-like annihilation, the volume increase of the PSB results in extrusions, interstitial-like annihilation to intrusions, respectively. Polák refined and modified the model in such a way, that is was possible to quantitatively calculate the rate of vacancy production in the PSB and their transfer from the PSB towards the matrix, eventually allowing for the prediction of the PSM height evolution as a function of the number of cycles, *dh/dN*. Intrusions within the PSMs are considered as crack initiation sites. Further growth of the fatigue crack is considered as short crack growth. Following the definition of Suresh and Ritchie, the short cracks are referred to as microstructurally short cracks as long as the crack size is comparable to the characteristic microstructural dimensions.

Ms Mazánova presents a critical review about the current state of research with a focus on the phenomenological nature of short cracks and the possibilities to apply standard fracture mechanics approaches for life prediction, like the stress intensity factor (ΔK) or the J-integral concept, respectively. Following an approach suggested by Pólak and co-workers, the crack growth rate *da/dN* can be correlated to the plastic strain amplitude ε_{ap}. Today, much modelling effort has been directed to the correlation of the crack propagation rate with the local microstructure and microstructural barriers. As a minor shortcoming of the thesis, the corresponding oscillating propagation rates that prevail within the microstructural short crack regime has been left out to some extent.

As suggested by the title, a substantial part not only of the experimental work is about high-temperature fatigue loading of SANICRO25. However, only one page of the section "current

Head of department: Univ. Prof. Dr.-Ing. habil. Ulrich Krupp Tel: +49 241 8092913
E-Mail: krupp@iehk.rwth-aachen.de Fax: +49 241 8092224

state of the knowledge" is dedicated to the respective differences in cyclic plasticity and fatigue damage mechanisms.

To conclude, it should be mentioned that the "current state of the knowledge section" fits smoothly to the "discussion" section, where additional references to recent literature work is given.

2 Experimental Work and Results

Ms Mazánova carried out strain-controlled cyclic testing experiments on polished specimens of SANICRO25, where she used a light-optical microscope for in-situ-tracking of PSMs, fatigue crack initiation and propagation on the specimen's surface. To limit the area of crack-initiation sites, the specimens were given a shallow notch. Electropolishing of the surface prior to cycling allowed for EBSD analysis (electron back-scatter diffraction) of the crystallographic orientation of those grains involved in the crack initiation process.

To my opinion, the most careful investigation of the persistant slip markings (PSM) using focussed ion beam milling (FIB) for in-depth analysis and thin foil lift-out followed by transmission electron microscopy (TEM) is the most impressive part of the thesis. It was proven that the PSMs being visible at the specimen's surface are a result of localized slip within persistant slip bands (PSBs). However, no characteristic ladder-like structure of the PSBs was found, which was attributed to the more complex dislocation characteristics in the high-alloyed SANICRO25. To my knowledge, the mechanism of PSB/PSM formation has not been analysed in combination with crack initiation and propagation statistics before. Ms Mazánova could give convincing evidence of a twin-boundary crack initiation mechanism that was postulated before by Neumann and later by Blochwitz and Tirschler, i.e., due to elastic anisotropy cracks initiate at every second twin boundary and continue growing by alternate operation of slip systems rather than along the twin boundary. At higher applied strain amplitudes ε_a, a certain degree of intercrystalline crack initiation was observed and attributed to PSMs emerging at grain boundaries and eventually leading to interfacial decohesion.

Since SANICRO25 has been developed for applications at elevated temperatures, a second part of the thesis deals with the mechanisms of cyclic plasticity and environmentally assisted crack initiation/propagation at 700°C. A thorough oxidation analysis revealed a pronounced attack of the grain boundaries, which were shown by FIB milling as to act as intergranular crack initiation sites. The intergranular oxidation products were identified as Fe-rich outer oxides and Cr-rich inner oxides by STEM-EDS. In addition to that small $M_{23}C_6$ precipitates were found and analysed by HRTEM (high-resolution TEM). These carbides are considered as to be formed during high-temperature cycling; however, this was not proven in a quantitative way. The initial state of SANICRO25 was studied by TEM, where only Nb-rich Z phase precipitates were identified. Since the material was solutionized at 1200°C and air-cooled, carbide precipitates were not expected.

After initiation at the grain boundaries, fatigue cracks tend to propagate perpendicular to the loading axis, governed by multiple slip at the crack tip. Although the thesis contains beautiful EBSD results on the crack path into the depth, no quantitative conclusions were drawn. From a technical point of view, the influence of dwell times at maximum tension on the continuation

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E-Mail: krupp@iehk.rwth-aachen.de Fax: +49 241 8092224

of intergranular cracking and the correlation with the structure of the respective grain boundaries would have been interesting aspects of research.

3 Discussion / Contribution to the Knowledge of Engineering Science

The main result of Ms. Mazánovas thesis is the correlation of planar dislocation plasticity concentrated in PSBs with the occurrence of PSMs at the specimen's surface. The PSMs can be considered as the prevailing prerequisite of crack initiation during cyclic loading. The experimentally found statistics on crack initiation and propagation are nicely discussed in terms of the crystallographic orientation, i.e., the Schmid factor of the respective grains. The transition from stage I crystallographic crack propagation to stage II normal-stress-controlled crack propagation depends on the plastic strain amplitude ε_{ap} and is often limited to only very few grains. Ms Mazánova tried to implement her most convincing micromechanical results into simplified continuum mechanics models, where the crack propagation rate (in the case of short cracks strongly governed by the local microstructure) is correlated with the remote stress or plastic strain amplitude. For the transition to long cracks this approach might be meaningful; here, in spite of the large scatter of *da/dN* data, a reasonable agreement was found using the equivalent crack length concept and the plastic strain amplitude ε_{ap} as crack driving force (according to Polák's model).

In summary, Veronika Mazánova provides an important contribution to the understanding of cyclic plasticity at both, room temperature and elevated temperature, respectively, that is of high relevance for further material development and mechanism-based life prediction. The results are critically discussed, clearly showing the limits of the applied experimental and theoretical approaches.

4 Presentation of the Thesis

The thesis is nicely presented following a scientifically formal structure, i.e., (i) introduction and aims of the work, (ii) a review of the current state of knowledge that is put into a historic context, (iii) a clear introduction of the experimental methods, (iv) results, (v) critical discussion, (vi) conclusions, and (vii) references. Although some of the analytical and numerical modelling work on the interactions between local microstructure and the corresponding crack propagation rates has not been mentioned, it was a pleasure to read the well-illustrated thesis that provides very impressive and significant insight in the nature of cyclic plasticity and fatigue crack initiation. There are a few spelling errors; the respective non-mandatory comments/corrections are included in the referee's copy of the thesis and will be given directly to the candidate. All graphical representations and micrographs are of high quality; they are chosen and explained in the text in such a way that they support the reader in following and interpreting the results.

Beside the thesis, a number of 10 peer-reviewed journal papers and 6 conference papers, authored or co-authored by Veronika Mazánova, support the fruitfulness, the high scientific relevance and the substantial value for ongoing research of her work.

Head of department: Univ. Prof. Dr.-Ing. habil. Ulrich Krupp Tel: +49 241 8092913
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6 Final Statement

According to my opinion, the thesis is acceptable without major corrections. Its scientific content and quality justifies the candidate, Veronika Mazánova, to be granted the PhD academic degree after having successfully defended her thesis.

Aachen, 19 November 2019

(Prof. Dr.-Ing. Ulrich Krupp)

Head of department: Univ. Prof. Dr.-Ing. habil. Ulrich Krupp Tel: +49 241 8092913
E-Mail: krupp@iehk.rwth-aachen.de Fax: +49 241 8092224