

Use of femtosecond laser spectroscopy for micro-crack analysis

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ABSTRACT. *It is well known that the initiation and propagation behaviour of small cracks is strongly influenced by the microstructure. In addition to the visible structure on the surface the information of 3D microstructure is essential for the evaluation of influencing factors. Investigations of micro-crack behaviour have been performed with intermetallic γ -based TiAl. The material selected is characterised by lamellar colonies of hard α_2 - and relatively soft γ -phase as well as by ordered B2-phase along colony boundaries. Starter notches facilitate systematic studies as cracks initiate from notch tips. In this case artificial notches in the scale less than colony dimensions were made by the femtosecond pulsed laser technique, which causes no significant damage in the vicinity of the notch. After multistage tensile compression test under increasing load several small cracks were generated and analyzed in SEM. Combining the femtosecond laser technique with a ICCD- spectrograph it was possible to analyze the surrounding microstructure of these micro-cracks and their propagation in depth direction by successive abrasion.*

INTRODUCTION

The fatigue damage accumulation of metal-based materials can be divided in three parts. At first micro-structurally short cracks are initiated due to micro-plasticity which then extend until they reach the size of so-called physically small crack. Both stages of crack growth are strongly influenced by the microstructure. The final stage of the fatigue damage accumulation process comes about when the worst crack starts to extend towards its critical size. In this stage the time to failure can be described by linear elastic fracture mechanics (LEFM) [1]. In many cases micro-crack initiation and propagation take most of the lifetime. Consequently the damage mechanisms in this regime have to be well understood for safe living. A systematic study of small crack extension is rather time-consuming due to the numerous influencing factors which govern the crack initiation stage. Hence it is quite hard to get well-defined starting cracks. An alternative may be to use artificial micro-notches, e.g. made using focus ion beam (FIB) technique.

Another approach with more rapid machining rate is given by femtosecond pulsed laser techniques. It was shown [2, 3] that micro-cracks are initiated from these artificial notches under cyclic loading. Using the fs-laser it has to be made sure that the heat affected zone formed by heat diffusion during processing is negligible and that the cracks initiated from these micro-notches possess extension mechanisms similar to those of natural small cracks [2].

Small cracks can propagate both intergranularly or transgranularly. A mixture of both extension modes can be observed in engineering materials with complex microstructure [4], where sometimes a transgranular crack path is observed on the surface and the same crack extends along phase boundaries in an intergranular mode or vice versa. Therefore a three-dimensional analysis of the crack path is crucial in understanding micro-crack growth. New insights in this problem may be gained by using the fs-laser technique again. A photomultiplier (PMT) detector combined with laser-induced breakdown spectroscopy (LIBS) allows signal strength analysis and quantitative analysis of chemical elements, respectively. Successive ablation of the material near a crack using both technologies can provide a useful tool for examining three-dimensional crack propagation.

MATERIAL

Investigations of paths of micro-cracks were performed with an intermetallic γ -TiAl alloy. Because of their good specific properties up to 700°C this material class is promising candidate for structural components of turbines and combustion engines. Conventional γ -TiAl alloys consist primary of colonies with lamellar arranged hard α_2 -phase (Ti_3Al , hexagonal DO_{19} structure) and relatively soft γ -phase (TiAl, tetragonal L1_0 structure). Optional heat treatment causes segregation of globular γ -grains and leads to a duplex or near lamellar microstructure.

There are numerous studies on the influence of microstructure on crack initiation and crack growth in lamellar titanium aluminides. The experiments show [5-9] that micro-cracks are initiated within the colonies in directions parallel to lamellae. The angle between the initiation plane and the loading direction is either around $90\pm 15^\circ$ or $45\pm 15^\circ$. This is in agreement to the planes of maximum tensile and shear stresses, respectively, on lamellar interfaces, where the cohesive force is expected to be low. In the ensuing extension phase the crack may propagate in a translamellar or an interlamellar way or it can extend along colony boundaries. In all cases Mode-I is the most likely crack propagation mode. Cracks can be retarded if they have to cross lamellae as more energy is needed for transgranular crack extension than for the intergranular one. A crack can be stopped, if it encounters lamellae with an orientation which deviates strongly from the crack plane.

In this study the relatively new texture-free β -solidifying cast TNM alloy with following chemical composition Ti-43Al-4Nb-1Mn-0,1B (at.%) was used. Figure 1 presents the etched microstructure of this alloy. Lamellar colonies have the size of 50-500 μm and typical lamellae thicknesses up to 2 μm . Distinctive colony boundaries

comprise globular γ -grains and the third β -phase (bcc solid solution or B2 ordered variant). Existing porosity and Ti-boride segregations are a consequence of casting without Hot Isostatic Pressing (HIP) and presence of Boron, respectively.

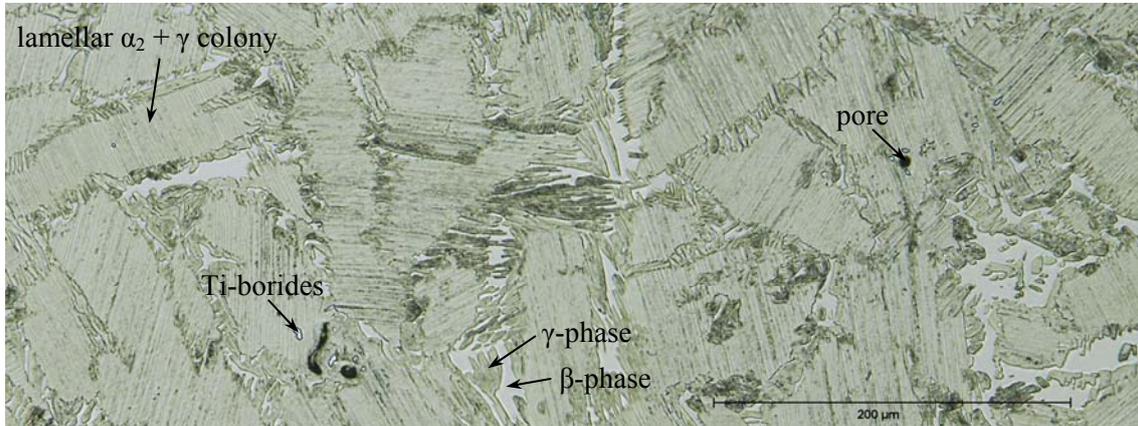


Figure 1. Microstructure of the Ti-43Al-4Nb-1Mb-0,1B alloy

The result of a quantitative chemical analysis using energy dispersive X-ray technique (EDX) are summarised in Table 1. It should be noted that analysis of γ - and α_2 -phases were done on wide lamellae. However the results can be somewhat uncertain as Monte Carlo simulations of electron transport in such phases show that the interaction volume can exceed the lamellae size. Nevertheless EDX measurement results from literature [10-13] on globular structures exhibit comparable values. Much more precise measurements with three dimensional atomic probe (3DAP) microscopy on lamellae [14] show slight differences in the chemical composition of α_2 -phase with a ratio of 59,20 : 32,41 at.% (Ti:Al).

Table 1. EDX results of chemical composition major phases (at.%)

Phase/Segregation	Ti	Al	Nb	Mo
γ	51,13	43,51	4,28	1,08
α_2	55,91	38,95	4,18	0,96
β	57,09	34,68	5,20	3,04
Ti-boride*	83,43	1,59	12,76	2,22

* Boron is not listed because of its low atomic weight and consequently high measurement errors

FEMTOSECOND PULSED LASER TECHNIQUE

The femtosecond laser system consists of a Ti:Sapphire fs-oscillator (Femtosource Scientific Pro, Femtolasers) and a Chirped-Pulse Multipass Amplifier (Femtopower Pro,

Femtolasers). Pulses of $500\mu\text{J}$ with a duration of 35fs and a wavelength of 800nm are generated at a repetition rate of 1kHz. These were coupled to a modified microscope using a mirror system. There is a coupling mirror within the tube of the microscope which allows to change between observation mode (for specimen positioning and observation) and fs-pulse mode. The positioning accuracy of the system is 10nm. Figure 2 gives an overview of the femtosecond laser system.

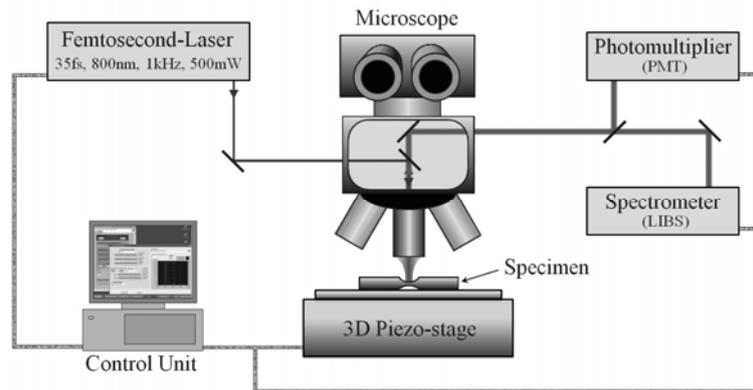


Figure 2. Schematic illustration of femtosecond laser system

The amount of energy of the fs-laser system which is transferred to the specimen surface in the focal point is sufficient to transform $0,5\text{-}5\mu\text{m}^3$ of the material into the plasma state. The luminescence of the plasma possesses characteristic emission lines which allow identifying elements. Hence it is worthwhile to perform a spectral analysis using a ICCD camera. The intensity of these emission lines can be related to the number of atoms of a specific element in the plasma and is a convenient way to determine the local chemical composition of the material in question. This may allows also identifying different phases in a specific material. The method sketched here is known in the literature as Laser induced Breakdown Spectroscopy (LIBS) [15-16].

Another application of the LIBS method in materials engineering is the analysis of crack paths in the early stages of crack initiation or growth. The signal will be reduced significantly when crossing a crack as the number of atoms in the analysed volume element is diminished. This idea is part of an ongoing project.

EXPERIMENTS

Micro-cracks were initiated in smooth specimens under stepwise increasing loading and under fatigue loading. The first test was performed with a smooth flat specimen with an hourglass notch. The load train started with a tension-compression load cycle with a small load leading to a stress level of 50MPa in the smallest cross section of the specimen. The maximum load was increased in steps in the following cycles. First deviations from linear elastic behaviour were detected at a stress level of 400MPa. The

specimen fractured at a maximum load of 650MPa. Several micro-cracks were detected in the vicinity of the fatal crack which were analysed in more detail using LIBS spectroscopy.

The second test was a regular load-controlled push-pull fatigue test ($R=-1$) with a cylindrical specimen. Smooth notches were introduced on two sides which allowed to observe the specimen surface during the test with the help of a travelling long-distance optical microscope. A strain gage was attached to the specimen on its reverse side. Fracture occurred at 151.062 load cycles. The strain gage indicated that there was some damage accumulation in the gage section even though the fatal crack was initiated somewhat outside at the top of the notch and no other cracks could be detected. Figure 3 shows how the fatal crack was first initiated from a large pore, then had a certain amount of microstructure controlled growth and reached a size just below its critical size at 150.000 load cycles. A detailed analysis of the pore showed that it had a maximum diameter of about $65\mu\text{m}$.

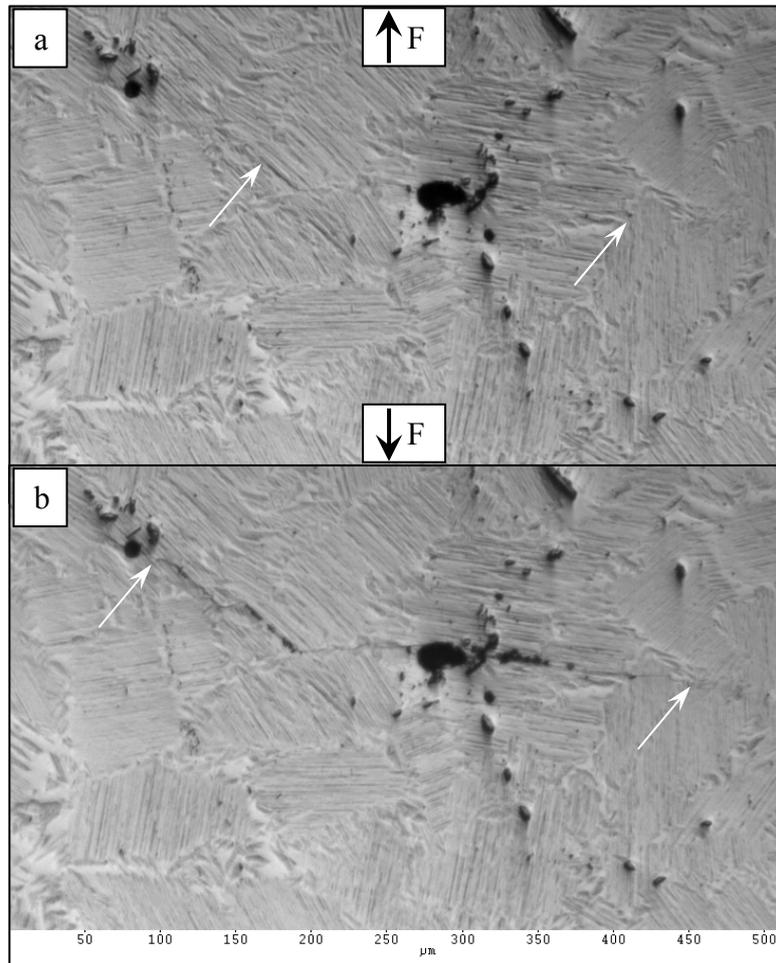


Figure 3. Crack initiation and propagation in TiAl-alloy depending on microstructure (a: 70.000 load cycles, b: 150.000 load cycles)

A third test was performed using artificial notches introduced at well-defined locations in the microstructure, see Figure 4. The size of these notches was defined on the basis of the results of the second test, as the artificial notches have to be larger than the fatal natural flaws for obvious reasons.

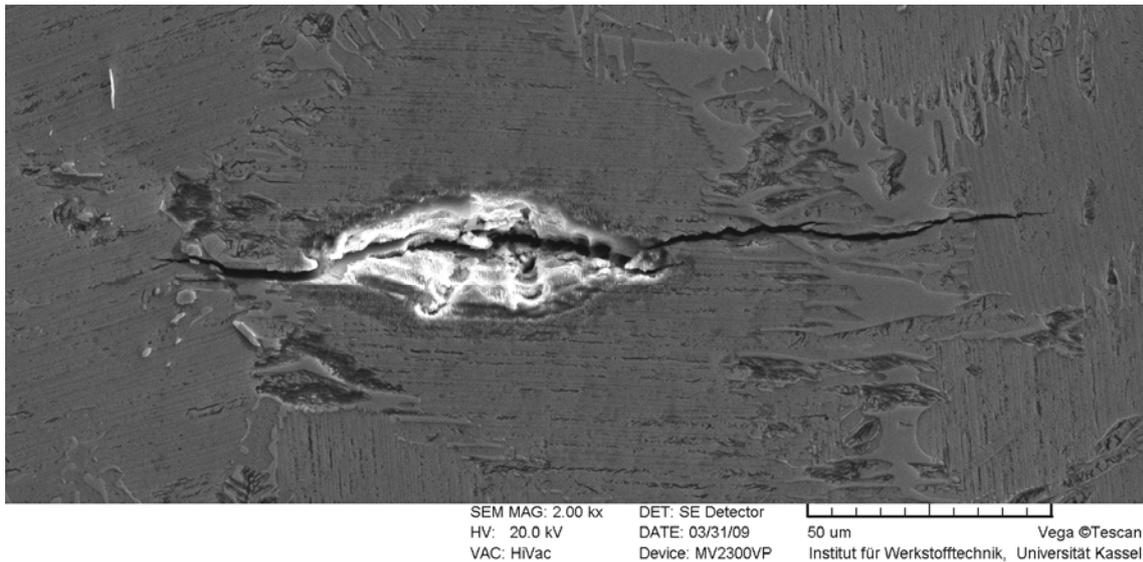


Figure 4. Micro-crack initiation from artificial notches

RESULTS

During the fatigue test a micro-crack was initiated from the notch in Figure 4 which first propagated in an interlamellar mode. This is also true for the propagation in depth direction. After crossing the intercolonial phase it continues in a pure mode-I propagation mode.

Crack path analysis using LIBS was performed on the secondary cracks found in the first test. Figure 5 shows that the LIBS method can easily detect cracks. A crack path analysis is possible even in depth direction. Correlating these pictures will yield the three-dimensional contour of the crack. Preparation time for each of the LIBS pictures is 2:28 minutes. This implies that a micro-crack with a depth of 50µm can be analysed within 2:06 hours, i.e. in a very small time compared to FIB-analysis or the synchrotron radiation method. The disadvantage of the method is its reduced resolution power. However, a better fine-tuning of parameters of the laser system to the material is feasible and is anticipated to give improved results.

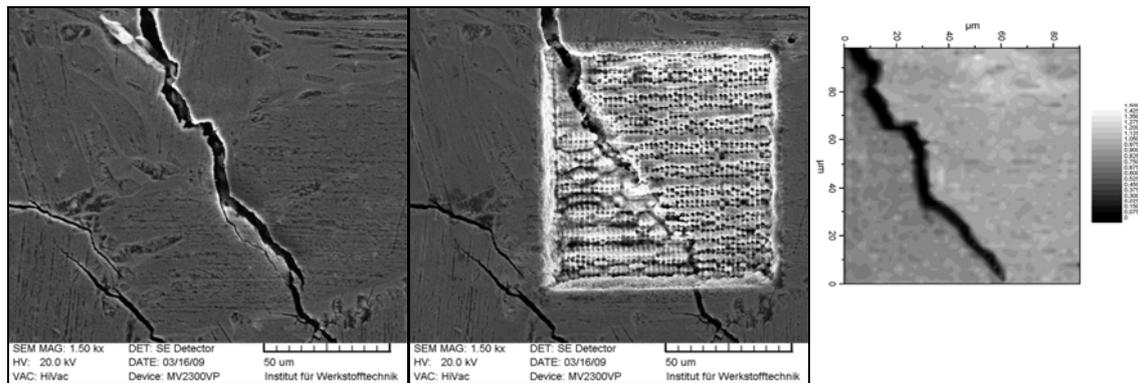


Figure 5. a) SEM picture of a secondary micro-crack, b) SEM picture of a secondary micro-crack after fs-LIBS analysis, c) results of fs-LIBS during the first ablation.

Further investigations of micro-crack initiation from the micro-notches are under way.

CONCLUSION

Femtosecond laser spectroscopy is a promising tool to analyse micro-cracks. The preliminary results show that Laser induced Breakdown Spectroscopy (LIBS) can be employed to study the crack path in depth direction in a very efficient way. This will help to understand the extension mechanisms of small cracks and their interaction with microstructure.

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