Atomic force microscope studies of the local slip at loaded crack tips in NiAl
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Abstract

The scanning probe microscopy has great advantages over other high-resolutional techniques like transmission electron microscopy in that no extensive specimen preparation, such as thinning, is required. This is important for examinations of crack tips, since thin film effects obscure the deformation behavior of cracks. For the first time, the concurrent processes of crack tip blunting by dislocation emission and unstable crack propagation were studied systematically with the atomic force microscope (AFM). Brittle cracks were initiated in NiAl single crystals. NiAl serves as a model alloy for the mechanical behavior of intermetallic compounds. A small bending device was constructed in which the specimens were loaded stepwise to measure the displacement fields of the crack tip ‘in situ’ at different load levels. With this loading device cracks were propagated in small steps of a few micrometers. From the AFM images dislocation distributions were obtained as functions of the applied load. Nearly radial symmetric elastic deformation fields were observed at the crack tip with a maximum depth of 46 nm. In addition, material parameters as the fracture toughness $K_{IC}$ were calculated from the crack tip opening at the onset of brittle crack growth. The measured value of 1.5 MPa√m compares favorably with results from standard fracture tests.

1 Introduction

From the theoretical point of view, many crystalline solids have unexpectedly high fracture toughness values, but still fail by unstable cleavage. The loaded crack emits dislocations which shield the crack tip from the applied load but if the local stress exceeds the cleavage stress, the crack becomes unstable. This local stress depends on the dislocation distribution. Since dislocation nucleation and glide is thermally activated, this distribution, and therefore the brittle-
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to-ductile transition, depends on loading rate and temperature. However, the experimental verification of models\textsuperscript{2,3} which describe the brittle-to-ductile transition is difficult. It requires the measurement of the growth of the plastic zone with a resolution on a nanometer scale.

The scanning probe microscopy techniques (SPM), such as scanning tunneling microscopy (STM) and atomic force microscopy (AFM) have turned out to be powerful tools for atomic-scale surface characterization\textsuperscript{4} and should be potential tools for these measurements. Compared with other high-resolution microscopes, like the transmission electron microscope, the measurements can be done on bulk specimens. This is an advantage, since in thin films the strong interaction between crack tip, dislocations and free surfaces and the unknown stress state at the tip complicate the interpretation. Only little experimental work has been done to study brittle cracks systematically with SPM\textsuperscript{5,6,7,8}.

For atomic resolution of crack tips in metals the experiments have to be performed in UHV to prevent oxidation of the surface. Since there is only one crack tip on the specimen surface the SPM must be able to inspect any detail within a relatively large area, hence 3-dimensional positioning is absolutely necessary\textsuperscript{9}. In addition the brittle crack should be kept under load to measure the displacement fields and dislocation distribution at different load levels. Since the surface preparation of NiAl in UHV is difficult, we restrict ourselves in this work on measurements with an AFM at ambient air.

2 Experimental

AFM measurements of the surface topography were performed with a Nanoscope III AFM from Digital Instruments. The force sensor, consisting of a Si\textsubscript{3}N\textsubscript{4}-cantilever with a tip radius of about 30 nm, gave a lateral resolution of nearly 2 nm and a vertical resolution of 0.2 nm. Taking a picture of 512 x 512 points took nearly 1-10 minutes. In addition, needlelike microtips which were

![Figure 1: Specimen types, dimensions, and orientations of NiAl single crystals.](image-url)
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grown by electron beam deposition on the end of the pyramidal tip were tested. Image analysis and data processing was done mainly with an image processing system on an IBM compatible PC programmed in C++ by the first author.

Two types of specimens were cut by spark erosion from stoichiometric NiAl single crystals grown by a modified Bridgman techniques from high-purity elements:

- Compact tension (CT) specimens with a height of 1-2 mm and a width of 10 mm (Figure 1).
- Four point bending specimens with a length of 13 mm and a thickness of 2 mm (Figure 1).

All specimens were oriented in the so called „soft“ orientation. The specimens were notched by spark erosion and the pre-crack on a {110} fracture plane was made by pressing a sharp knife in the bottom of the notch initiating a pop-in of a length between 50 and 100 μm. All specimen dimensions fulfilled for NiAl at room temperature the ASTM standards for testing brittle materials. The specimens were loaded in a small bending device, small enough to be put into the AFM.

Figure 2 shows this device. The samples were pre-loaded by turning a fine torque-controlled screw. Further loading inside the AFM was done by a special piezostack, which allowed a maximum extension of 3 μm at an voltage of 500 V (round block on the left side). With this piezo a force of 220 N could be reached which was sufficient to fracture the specimens. A similar device was built to load small CT specimens.

Figure 2: Device to load bend specimens continually and in situ inside the AFM.
Table 1: Roughness values of NiAl after different preparation steps compared with semiconductors

<table>
<thead>
<tr>
<th>Material</th>
<th>Preparation Method</th>
<th>rms-Value</th>
<th>Lateral Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>NiAl single crystal</td>
<td>only grinded (4000)</td>
<td>14 nm</td>
<td>≈ 60 nm</td>
</tr>
<tr>
<td>NiAl single crystal</td>
<td>polished 4 m diamond paste</td>
<td>6.5 nm</td>
<td>≈ 60 nm</td>
</tr>
<tr>
<td>NiAl single crystal</td>
<td>polished 1 m diamond paste</td>
<td>1.4 nm</td>
<td>≈ 60 nm</td>
</tr>
<tr>
<td>NiAl single crystal</td>
<td>electropolished with perchlorid acid</td>
<td>2.4 nm</td>
<td>5 - 100 nm</td>
</tr>
<tr>
<td>GaAs-Wafer</td>
<td>polished</td>
<td>0.35 nm</td>
<td>5 - 30 nm</td>
</tr>
<tr>
<td>Si(111)-Wafer</td>
<td>polished</td>
<td>0.26 nm</td>
<td>5 - 20 nm</td>
</tr>
</tbody>
</table>

The investigations described above require a low surface roughness, to distinguish cracks and dislocations from ordinary preparation defects like polishing traces. This is clearly seen in Figure 3 which shows that it can be difficult to follow the crack very close to the tip because of the large amount of polishing traces. Therefore different preparation techniques were tested to minimize the roughness. Results of this are summarized in Table 1. The roughness was checked calculating the rms roughness
\[
\sqrt{\frac{1}{n} \sum_{i=1}^{n} (z_i - \bar{z})^2}
\]
from AFM images with a scansize of 4 μm. For the mean lateral size of the roughness objects we obtained approximate values from the autocorrelation function\(^{14}\). The best rms values for NiAl specimens after grinding and polishing with diamond paste are

Figure 3: AFM image of a mode I crack in a NiAl specimen with a rms-roughness of 2.4 nm. The crack starts at the lower left side of the image, but it is difficult to determine the position of the crack tip because of the existence of large polishing traces.
in the range of 1 - 2 nm. Electropolishing did not improve this values significantly but there the surface structure changes to a more hilly like topography with smaller patterns. In comparison, semi-conductor surfaces with a rms-roughness of only 0.3 nm can be easily polished.

Since every SPM image is the result of convoluting the surface topography with the tip profile, using of well known and sharp probe tips is important for imaging cracks. As long as very flat and smooth surfaces are scanned these effects can be neglected. For examining rough surfaces (Figure 3) deconvolution of the SPM data should be performed\(^5\), which is only possible if the tip shape is well known. A mode I crack opening component (Figure 4) can be screened completely by a blunt tip if the crack is also opened with a considerable amount in mode III\(^7\). Therefore different probe tip shapes were used to examine the crack profile at several crack positions. Figure 5 shows the result of these measurements. Imaged with a standard Si\(_3\)N\(_4\) cantilever the crack which extends through the whole specimen seems to be only 150 nm deep. Using an Electron Beam Deposition (EBD) tip with a length of 1 \(\mu\)m reveals at least a crack depth of 800 nm. But now the profile is disturbed through friction effects at the crack flanks. The effective probe tip radius can be estimated by fitting circles on these profiles at the edges of the crack walls, which should be oriented perpendicular to the specimen surface in the ideal case. These measurements imply a tip radius of 65 and 40 nm for the Si\(_3\)N\(_4\) tip and the EBD grown tip, respectively. Despite the considerable influence on the crack profile no improvement was seen by imaging the crack tip region with an EBD grown tip.
3 Results and Discussion

3.1 Dislocation emission at the crack tip

Figure 6 shows a mode III crack tip in a CT specimen. This crack was imaged with the AFM over a length of 50 μm by moving the visual field of the microscope manually. The image shows slip traces which emanate from the crack tip under an angle of 45° to the crack propagation direction. These slip traces can be interpreted to result from screw dislocations gliding on {100} slip planes. In a scanning electron microscope these shallow slip traces are barely visible. With the AFM, however, the height of the slip steps can be measured directly as a function of the distance from the crack tip. A special software routine subtracts the mean height from both sides of the glide step. Figure 7 shows this subtracted linescan. The height decreases from 15 nm directly at the crack tip.

Figure 6: AFM image of a Mode-III crack in NiAl. The crack runs from the lower left border of the image to the middle, where a glide step is emitted from the crack tip.
Figure 7: Schematics of the appropriated slip system for Figure 6 and distribution of glide step height as a function of the distance from the crack tip.

Figure 8: Two AFM images of the same crack tip at different load levels. The bright line running from the left border through the crack tip remains from polishing and has no correlation with the crack.

to zero at a distance of 25 μm. About 52 dislocations were emitted from the crack tip, taking a burgers vector length of 0.2887 nm. They are distributed in front of the crack tip with decreasing density. This behavior was observed with all CT specimens. Also loading of these specimens with a small loading device was performed. A detailed analysis of these measurements is given in another paper. No additional dislocation emission under load was visible, whereas the mode III crack opening component has grown clearly.

Another example of a similar crack is shown in Figure 8 for two different load levels. The corresponding increasing mode III crack opening displacement can be clearly seen as well as slip steps emanating from the crack tip. Further loading initiated unstable crack growth. A detailed analysis of the crack opening and glide step heights is given in Figure 9. The mode III component of crack opening displacement, $z$, is plotted as a function of the distance from the crack tip. Rising the load increased the elastic crack opening displacement, the
Figure 9: Mode-III component of crack opening z versus distance from the crack tip (compare with Fig. 4). Starting at the crack tip the glide step height distribution is shown.

plastic crack opening, however, taken as the slip step height at the crack tip, remained constant (15 nm). This means that no more dislocations were emitted. In addition Figure 9 shows the slip step height distribution which is the same for both AFM images. The distribution was steeper than that in Figure 7 and the slip lines had only a length of 5 μm.

3.2 Stable crack growth and fracture toughness

For pure mode I cracks (Figure 10), similar measurements as described above were performed in four point bending. These cracks could be loaded continuously inside the AFM with the loading device shown in Figure 2. Stable discontinuous crack growth was observed after loading with the piezo stack, as shown in Figure 11. The crack propagated in several steps with an individual step size of 1-3 μm. To load the specimen until ultimate fracture occurred the fine screw has to be tightened in between, since the extension of the piezo stack is limited to 5 μm.

The crack arrests by dislocation emission in the alternating slip orientation which results in a V-shaped crack tip. Such a typical V-shaped crack is shown in Figure 12. No glide steps appear on the surface, since the burgers vector of the blunting dislocations now lies parallel to the specimen surface. Only at the left side of the crack in Figure 12, a shallow glide trace could be observed which indicates a small misorientation of the {100} surface by 1°. Therefore the dislocation distribution in front of the crack tip cannot be examined from these measurements in the same way as in Figure 6. In ongoing work a different
localized damage specimen shape is used which allows measurements of slip step heights in four point bending specimens as a function of applied load. The results will be compared with dynamic computer simulations of the brittle-to-ductile transition in NiAl\textsuperscript{17}.

From Figure 12, the critical plastic crack opening displacement (COD), after which the micro pop-ins were produced, could be measured directly. The V-shaped part of the crack length which corresponds to the COD reached 85 nm on both sides of the crack. From this the fracture toughness $K_{IC}$ for initiating a pop-in can be calculated\textsuperscript{18}:

$$K_{IC} = \sqrt{COD \cdot E \cdot \sigma_y}$$

With a yield strength of 150 MPa and a Young's modulus of 188.1 GPa at room temperature\textsuperscript{12} this leads to a fracture toughness of 1.5 MPa√m which is in good agreement with theoretical and other experimental work. Bergmann et al.\textsuperscript{11} measured the fracture toughness by standard fracture tests but with larger specimens and obtained slightly higher values of 3 MPa√m. Theoretical calculations of the Griffith cleavage energy by Yoo et al.\textsuperscript{19} resulted in a fracture toughness of 1 Mpa√m.

Figure 10: 3D image of a mode I crack.
3.3 Elastic deformations at the crack tip

In a different representation of the above images elastic deformation fields at the surface are clearer visible. In Figure 13 only 7 discrete gray levels are used for the height scale of the measurements. So the border line between two gray levels corresponds to a line of constant altitude, i.e. constant elastic displacement \( u_z \) of the surface. Figure 13 shows these deformation fields at three different load levels, adjusted by the voltage of the piezo stack. Nearly radially symmetric displacement fields with a maximum depth of 46 nm developed under loading around the crack tip, because of the high stress concentration at the crack tip. This process is reversible by turning on and off the voltage at the piezo stack. This elastic deformation field can be used to measure local stresses at the crack tip. After discontinuous crack propagation as shown in Figure 11 the load level is reduced and also the corresponding deformation field. Under the assumption of a plane stress state at the surface, which exist typically in thin sheets, the elastic displacements \( u_z \) can be easily calculated from the stress fields at the crack tip:

\[
\frac{u_z}{d} = \frac{v}{E} \frac{K_I}{r} \sqrt{\frac{2}{\pi r} \cos(\phi/2)}
\]

where \( d \) is the specimen thickness, \( v \) Poisson's ratio, \( K_I \) the stress intensity factor, \( r \) the distance and \( \phi \) the angle at the crack tip.

Displacements calculated from this equation are much higher than the displacements measured by AFM. Several reasons are responsible for this: This

Figure 11: Crack advance after loading a four-point-bending specimen in-situ.
Figure 12: Stop and go crack growth: At about y = 1.1 μm the crack had blunted to a V-shaped tip by alternating slip. From the blunted tip the crack has propagated again by 0.78 μm (micro pop-in) and was stopped again by dislocation emission.

solution is only valid for atomically sharp, long cracks and not in the immediate neighborhood of the crack tip due to the singular behavior of the elastic stress fields. The state of stress at the surface of the four point bending specimens is not well described by the assumption of plane stress or plane strain. Therefore, three-dimensional Finite Element calculations of the displacements are necessary, to determine local stresses at the crack tip from the here shown AFM measurements.

4 Summary

In this paper it was demonstrated, that the AFM can be used as a new tool for examining local processes at crack tips. Examples are given, which show that dislocation distributions can be measured as a function of applied load in suitably oriented single crystals. From the slip step height as a function of distance from the crack tip the size of the plastic zone and number of dislocation within a slip plane can be estimated and correlated with the measured crack tip opening. With a small four point bending device constructed for in situ loading in the AFM, stable quasi-brittle crack growth was observed in NiAl single crystals. It was found that the crack propagated in micro-pop-ins. The critical COD for such a pop-in could be obtained directly from the AFM images and
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Figure 13: Elastic displacement fields, \( u_z \), at the crack tip under different load levels.

compared with the fracture toughness \( K_{IC} \). The calculated value of 1.5 MPa\(\sqrt{m}\) compares favourably with results obtained from standard fracture tests.

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References

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