Studies of Metallic Surfaces and Microstructures *with Atomic Force Microscopy*

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Atomic Force Microscopy (AFM) is capable of generating 3D images of surface topography with nanometer and Ångstrom resolution. This powerful technology allows material scientists new insight into the diversity of microstructures and defects. Applications of AFM to metal specimens allow quick and easy generation and quantification of images in the sub-micrometer and nanometer range. Reliable measurements of microstructural parameters are of great relevance for developing new alloys and for quality control in industrial production processes. No vacuum is needed for the atomic force microscope, and even measurements in liquids, such as diluted acids, are possible.

This application note describes how AFM provides the ability to study morphology, *in-situ* fracture processes, and plastic deformations on standardized specimens with resolutions that are unattainable with other measurement techniques.

Applications to Metallic Microstructures

Metallic structural detail covers many orders of magnitude in size. Therefore, different microscopic techniques like optical microscopy, scanning electron microscopy (SEM) and, for even higher resolution, transmission electron microscopy (TEM), are used for visualizing these microstructures. AFM is a very useful supplement to these techniques with scan sizes ranging from about 100µm down to several nanometers, comparable to and, in some cases, exceeding the very high magnifications achievable with TEM. This large dynamic range is illustrated in Figure 1 with four examples, where the microstructures of a perlitic steel, martensite, and a γ' precipitated nickelbase superalloy are resolved clearly with the AFM. Also, the oxidation behavior on an iron-chromium single crystalline alloy is shown, where small oxide grains with 20nm radius are crystallographically oriented on

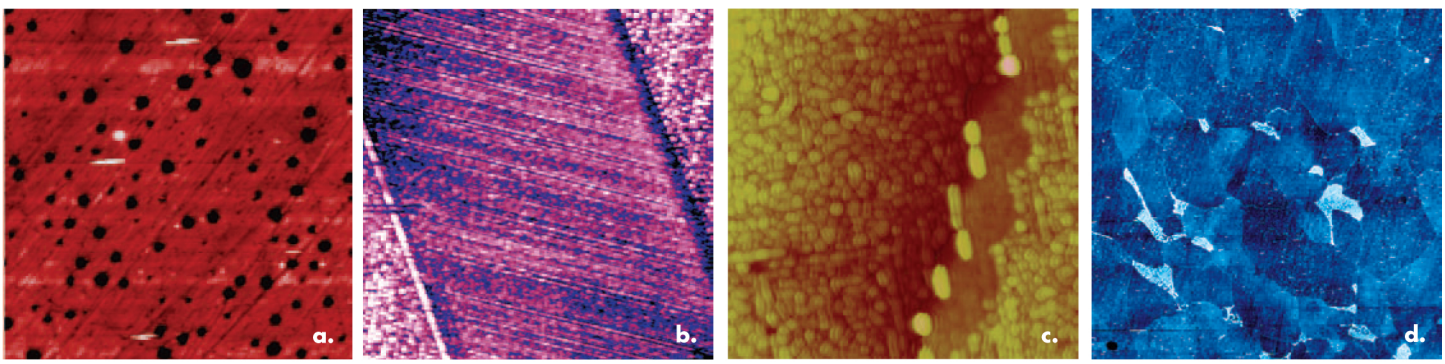


Figure 1. Contact-mode AFM height images of different metallic surfaces: (a) γ' precipitated nickelbase superalloy, 6µm scan, (b) martensite, 11.4µm scan, (c) Fe-Cr single crystalline alloy, 700nm scan, and (d) perlitic steel, 125µm scan. Brighter colors indicated taller features.

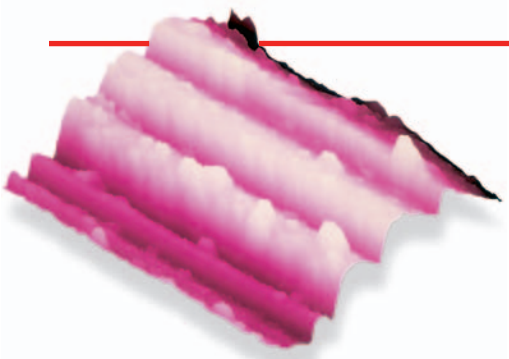


Figure 2. Brittle fracture surface of NiAl imaged with contact mode AFM. 5.7µm scan.

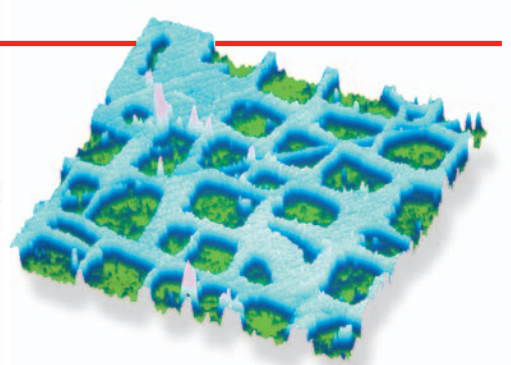


Figure 3. Microstructure of the superalloy CMSX-6. The cuboidal precipitates (green) correspond to the γ' phase. 4µm scan.

near-atomically-flat terraces. Here, the enrichment of chromium on the stepped surface prohibits oxidation in small bands on the upper side of the steps.

Surface Preparation and Roughness

Since AFM allows imaging of conductive and nonconductive surfaces directly in ambient air, surface preparation is generally an easy task. To image very small particles, however, etching and/or polishing is sometimes required to prepare a surface which is flat enough to resolve the particles (the mean remaining roughness of the surface must be kept lower than the particle size). The roughness itself is determined easily from AFM images. Some typical roughness values after different preparation steps are listed in Table 1.

Electropolishing is often the best choice for preparation of metallic specimens. Topographic contrasts, which are necessary for most microstructural investigations, can be developed by many chemical preparation methods as well. In comparison to these smooth surfaces, fractured specimens often show very high roughness levels. In Figure 2 an example of a brittle fracture surface is shown, where fracture facets with distinct orientations are resolved with the AFM.

Quantitative Evaluation of Microstructures

For exact quantitative measurements, the etching or preparation depth of the surface should be minimized – i.e. substantially less than the mean particle size. The superior vertical resolution of the AFM is optimal for these measurements. Figure 3 shows a 3D topographic image from a chemically-etched specimen of the superalloy CMSX-6. The lower (green) parts of the image corresponds to the γ' phase, which is responsible for the excellent high temperature properties of this alloy. Volume fraction and precipitation size obviously are important parameters for optimizing the high temperature performance of this alloy. A quantitative evaluation of such parameters is illustrated in Figure 4. The low etching depth (14nm) of the γ' phase is illustrated through the linescan. The area fraction of the precipitates was determined from a histogram of the image data, where two maxima reveal the microstructure of the γ' and matrix phases.

In other superalloys, spherical particles (instead of cuboidal) are precipitated through the heat treatment. Waspaloy, for example, shows a bimodal distribution of precipitates. Here, the small precipitate size prevents reliable quantitative evaluation of the γ' volume fraction with most microscopic techniques, and quantitative evaluation of TEM images is difficult, since determination of the transmitted film thickness is a time-consuming task.

Again, AFM overcomes these difficulties. Figure 5 shows an example, where the bimodal size distribution was determined from a single AFM image after an electropolishing procedure. Radii of 30nm and 200nm were found for the small and large precipitates, respectively.

Nanomechanical Studies

The AFM can also be used to indent the specimen surface plastically. Indentation techniques, as for example Vickers hardness testing, are well-known methods in the field of materials science for fast estimations of yield stress and tensile strength with hardness values calculated from optical inspection of the indentation size. With AFM, indentation techniques can now be performed at much smaller size and force scales.

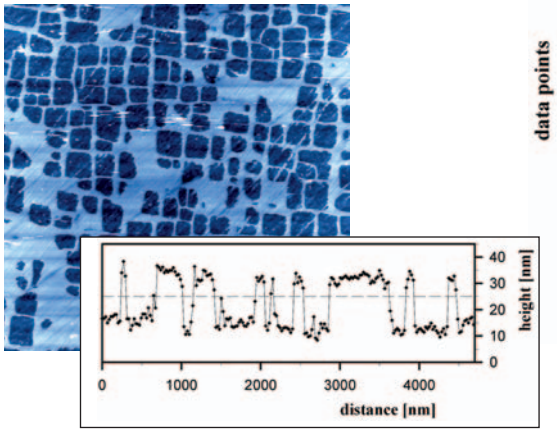
Specimen	Preparation Method	Roughness (R_a)
Fracture Surfaces	None	100nm-5µm
Steel	Mechanical polishing (1µm diamond paste)	2-5nm
NiAl Single Crystal	Mechanical polishing (0.25µm)	1nm
Superalloy	Chemical etching	3-4nm
Superalloy	Electropolishing	0.8nm
Microalloyed Steel	Electropolishing	0.6nm

Table 1. Typical roughness values for different metallic surfaces as measured with AFM.

For "nanoindentation," diamond tips are usually used on metal samples. The size of the indents are determined directly after indenting by scanning the affected surface with the same AFM tip that produced the indent(s). Nanoindenting is performed with maximum loads in the range of only a few hundred micro-Newtons, which also makes this technique very attractive for studying thin films. Figure 6 shows how the different phases of the superalloy CMSX-6 were indented with a diamond tip, and the different sizes of the indents reveal the lower hardness of the matrix phase. Indentation sizes can be reduced to ten nanometers or less, which makes possible highly localized studies of the mechanical properties of the microstructure (Figure 7).

In-Situ Loading and Investigation of Cracks

Another application of AFM is imaging of defects like cracks, dislocations and pores. The high resolution capability of AFM permits study of the processes of brittle and ductile crack growth in detail. To study such crack growth, a small specimen was mounted inside the AFM with a small force loading device which was built for the Digital Instruments MultiMode AFM. Preloading was done outside the AFM with a micrometer screw, and further loading inside the AFM occurred by applying an electric voltage to a small piezostack. Figure 8 shows two images from a loaded mode "I" crack in a single-crystalline specimen of NiAl. The crack grows after loading (from the left to the right image) in a quasi-stable brittle manner in steps of nearly 1µm length. In both images, the high-stressed red region around the crack tip was caused by small elastic displacements. The high stress intensity of the crack produces a small elastic depression zone (red) with a depth of only some ten nanometers. These elastic displacements around the brittle crack tip are best visible with the high vertical resolution of the AFM.



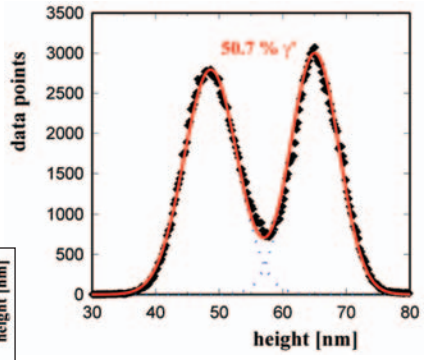


Figure 4. AFM image (top left) can be used to measure sections through the microstructure of the superalloy CMSX-6, 12µm scan. The height difference (14nm) between the two phases in CMSX-6 is obtained from a line section (left, bottom). The area fraction of the γ' phase is obtained from the histogram (above).

For larger specimens, Digital Instruments Dimension™ Series AFMs allow investigations with commonly used testing machines, where stresses and strains are applied and measured on tensile or even bend specimens. Figure 9 shows an example of a cracked specimen of NiAl with a fracture toughness (KIC) of 4 $MPam^{1/2}$ loaded under force control. The AFM image shows that even this material – which is extremely brittle at room temperature – shows small amounts of ductile deformation behavior, i.e. dislocation emission at the crack tip. Two glide planes, which include an angle of 45° with the crack, are activated through dislocation emission. The number and distribution of dislocations emitted from the crack can be counted by studying the kink motion of parallel polishing traces remaining on the surface after mechanical polishing. As a dislocation crosses a polishing trace, it will be kinked in a step corresponding to the Burgers vector length of the dislocation. From these plastic deformations and also from the elastic depression zone around the crack tip, the fracture toughness can be calculated. This work helps to develop a better understanding of fracture mechanism and the brittle-to-ductile-transition in metals and intermetallic compounds.

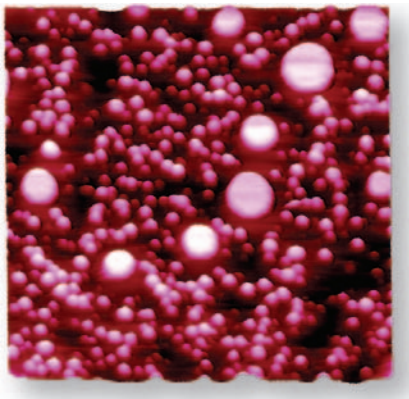


Figure 5. Waspaloy's bimodal precipitation distribution is responsible for its high temperature properties. The volume fraction of the small γ' precipitation and the particle size distribution can be determined from AFM images with high accuracy. 2.2µm scan.

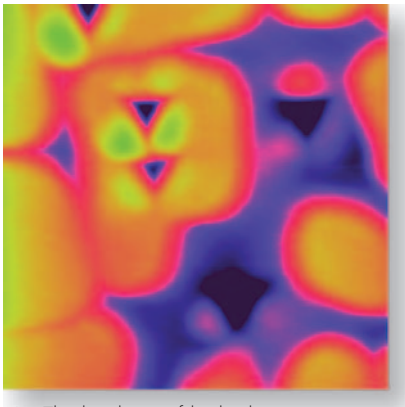


Figure 6. The hardness of both phases in CMSX-6 was compared through small nanoindentations obtained using a diamond probe tip. The smaller size of the triangular indents in the γ' phase (yellow) reveal it's greater hardness. 2µm scan.

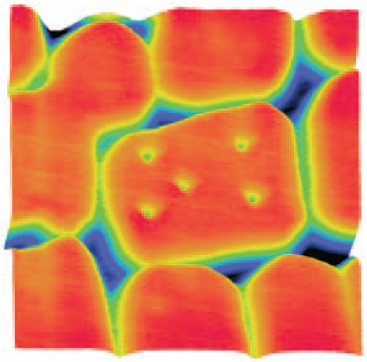


Figure 7. Nanoindents in the γ' phase of CMSX-6 produced with a maximum force of only 100μN. 1.5μm scan.

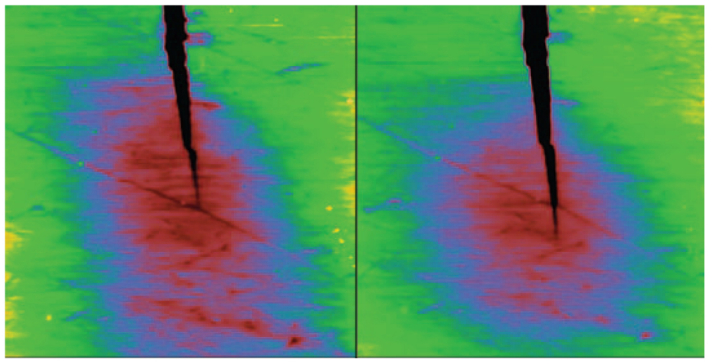


Figure 8. Brittle crack growth in the intermetallic compound NiAl. The red elastic depression zone around the crack tip visualizes the high stress intensity in this area. 8.6μm scans.

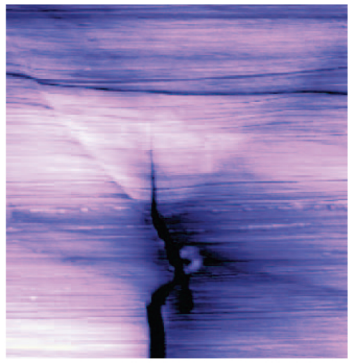


Figure 9. Crack tip with plastic deformation zone in NiAl. Horizontal polishing traces in front of the crack tip are kinked through dislocations emitted from the crack. 6μm scan.

Summary

Reliable, localized measurements of microstructural parameters are of great relevance for developing new alloys, and for quality control in manufacturing processes. AFM provides unique capabilities relative to other microscopies, including; superior resolution, 3D measurements, little or no sample/substrate preparation, and operation in ambient air or liquid. AFM also enables the study of the origin and the mechanism of plastic deformations, including fracture, on the micrometer and nanometer scales.

References

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