MICROSTRUCTURE CHARACTERIZATION AND IMAGING IN TITANIUM ALLOYS BY ATOMIC FORCE ACOUSTIC MICROSCOPY

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INTRODUCTION

Titanium is a strong, highly creep-resistant, light, heat-resisting, biocompatible, and corrosion resistant transition metal with a broad field of applications in industry [1]. The metal consists primarily of two different crystal structures: the hexagonal close-packed (hcp) α-phase and the body-centred cubic (bcc) β-phase. The bulk mechanical properties of titanium show big differences depending on the ratio of the two phases that have been formed during heat treatment. Pure titanium cooled slowly enough to allow diffusion will contain 100% α-phase at room temperature. Above the β transition temperature (BTT = 882°C for pure titanium) the bcc structure is stable. Depending on temperature and cooling rate the β-phase may either transform to the metastable β-phase or be replaced by stiff hcp α'- or soft tetragonal close-packed (tcp) α''-martensite structures [2]. The hcp α-phase shows the highest indentation modulus (~117 GPa), but exhibits a strong elastic anisotropy. The much less anisotropic bcc structure has a lower indentation modulus (~82 GPa for the metastable β-phase) [3]. The bcc structure contains more slip planes than the hcp structure and thus is easier to deform. Therefore and also to avoid substantial grain growth forming is preferably carried out just above the β transition temperature (BTT) [4]. Both phases can be stable even below the BTT if stabilizing elements are added to the titanium solution. Elements stabilising the α-phase as e.g. N, O, C, Al, Sn, and Zr (in decreasing order of α stabilization) increase the BTT. Elements as Fe, Bi, Mn, Cr, Co Mo, Ni, Pb, Cu, V, and Nb (in decreasing order of β stabilization) stabilise the β-phase and decrease the BTT [5]. The most commonly used titanium alloy is Ti-6Al-4V covering 60% of the titanium market in which 80% accounts for aerospace industry [5]. The BTT of Ti-6Al-4V with α stabilising aluminium and β stabilising vanadium, respectively, is about 1000°C [6]. While heating, the volume fraction of the β-phase increases. Since the total amount of β stabilising elements is fixed, the volume fraction of stabilizing elements in the β-phase decreases with increasing temperature. Therefore, when cooling again to room temperature, the β-phase becomes unstable and transforms into stable β- and secondary α-phase if there is time enough for diffusion. If the alloy is quenched, so that no time will be for atom arrangements the β-phase will either remain in its metastable state or transform into soft α''- (below ~900°C) or stiff α'-martensite. At room temperature, the β-phase is only stable when it is enriched with 15 wt.% vanadium. This can be obtained by slow cooling and/or annealing below 750°C. Depending on the heat treatment the α- and β-phases form different microstructures and arrangements. The structures are classified into three categories: lamellar, equiaxed, and a mixture of both [5].

Elastic properties and shape of the different phases forming the microstructure of a material determine its macroscopic behaviour. Thus quantitative characterization of microstructures in titanium alloys having undergone different heat treatment processes will provide with guidelines for materials design. Atomic Force Microscopy (AFM) [7] in combination with ultrasound is the near-field acoustic microscopic method Atomic Force Acoustic Microscopy (AFAM) [e.g. 8-12]. This technique enables to image and measure local elastic properties of sample surfaces with a spatial resolution in the nm range and hence is a convenient tool for this purpose.

EXPERIMENTS

A series of Ti-6Al-4V alloy specimens were heat treated at different temperatures and cooled to room temperature again with various cooling rates in order to obtain different microstructures with different amounts of α- and β-phase, and α''-martensite. Atomic force acoustic microscopy (AFAM) was successfully applied for qualitative imaging of the microstructural features of these samples. To verify the results and to distinguish between the two primary phases (α and β), backscattered electron (BSE) imaging and energy-dispersive X-ray spectroscopy (EDX) analysis were carried out. Objective of these investigations was to find out if only the ratio of α- and β-phases or additionally also changes in chemical composition and elastic behaviour of the single phases influence the macroscopic modulus of Ti-6Al-4V. Furthermore, a correlation of applied heat treatment and resulting microstructure was aimed for.

DISCUSSION OF THE RESULTS

Figures 1 to 3 show AFAM and BSE images of the different samples. The results are consistent, however, in contrary to BSE, the variation in stiffness between different grains of the α-phase due to its strong single crystal anisotropy and different lattice orientations are clearly visible (Figs. 1 to 3) in the AFAM images, and the very fine needle-like α''-martensite structure can be visualized by the AFAM technique (Figs. 2 and 3). The different treatments of the samples and the resulting microstructures are described in detail in the figure captions.

The EDX analysis revealed that the chemical composition and hence lattice parameters and elastic moduli of the α-phase are almost independent of the heat treatment whereas in the β-phase the amount of vanadium decreased with increasing applied temperature causing differences in the elastic moduli. Quantitative evaluation of AFAM measurements indicated an increasing indentation modulus of the β-phase with decreasing amount of vanadium. Ultrasonic bulk measurements, however, showed a decreasing macroscopic modulus with increasing temperature. These results clearly indicate that the predominant factor attributing to the change in modulus of Ti-6Al-4V is the volume fraction and not the chemical composition of the β-phase.

The martensite transition temperature was observed around 850°C. Samples quenched at this temperature attained the lowest indentation modulus since they contained the largest amount of the β-phase. Quenching at higher and also at lower temperatures leads to higher indentation moduli. Heat treatments above the martensite transition temperature followed by water quenching yield increasing moduli with increasing temperature because of martensite formation converging to a maximum value for treatments over the BTT (~1000°C) when specimens of 100% α''-martensite were formed. Samples annealed only below the martensite transition temperature contained an increasing amount of the α-phase with decreasing annealing temperature. AFAM as well as macroscopic ultrasonic measurements showed that the α-phase in Ti-6Al-4V is stiffer than the α''-martensite phase.

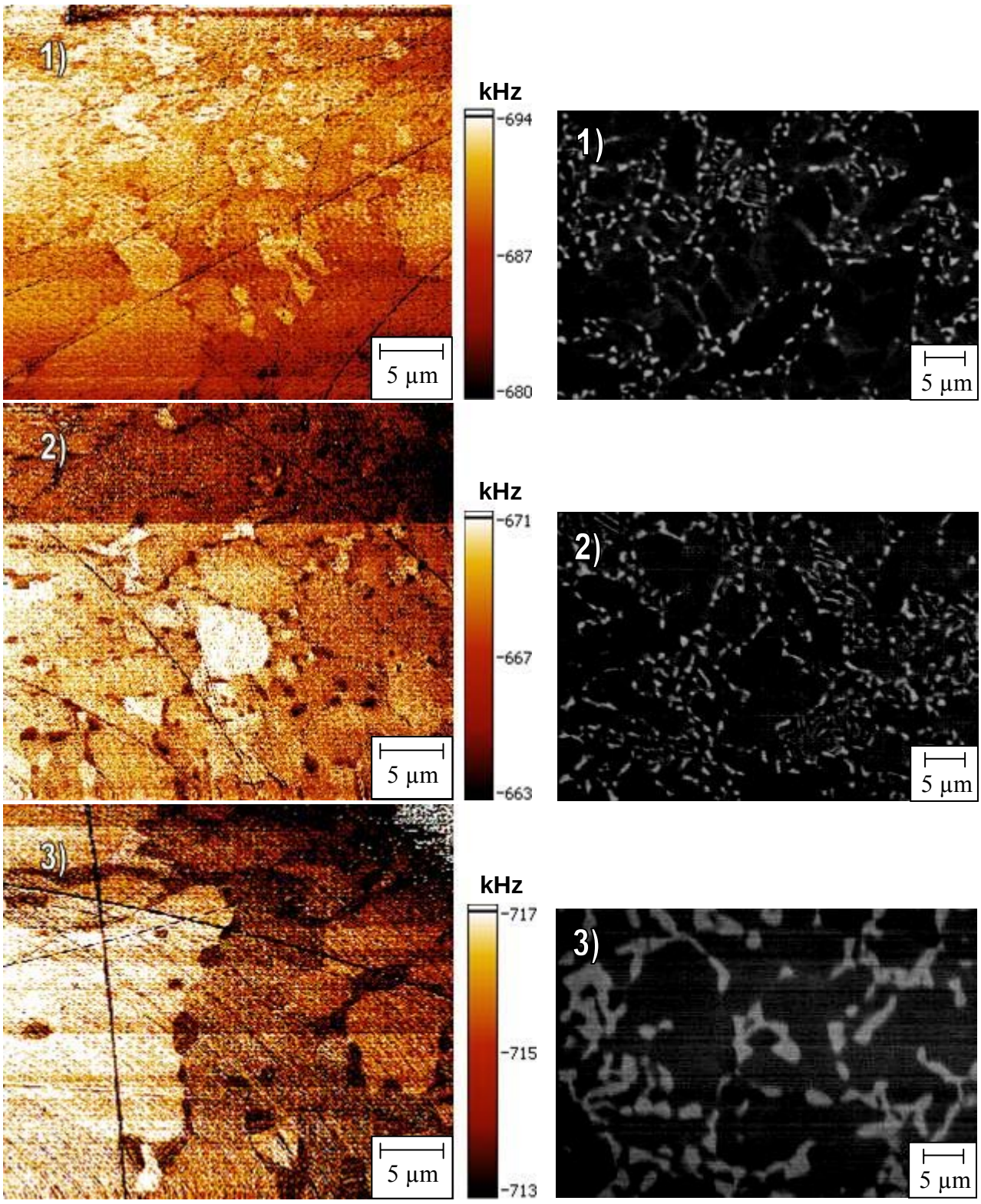


Figure 1. AFAM and BSE images of samples 1 to 3 (as received, for 1h heat treated at 650°C and at 850°C, respectively). From the BSE images the large grains are identified as α-phase. They show high contact resonances in the AFAM images indicating their higher stiffness compared to the β-phase appearing as small dark spots and regions between different grains of the α-phase. The variation in stiffness between different grains of the α-phase is due to different lattice orientations showing the strong anisotropic behaviour of α-phase.

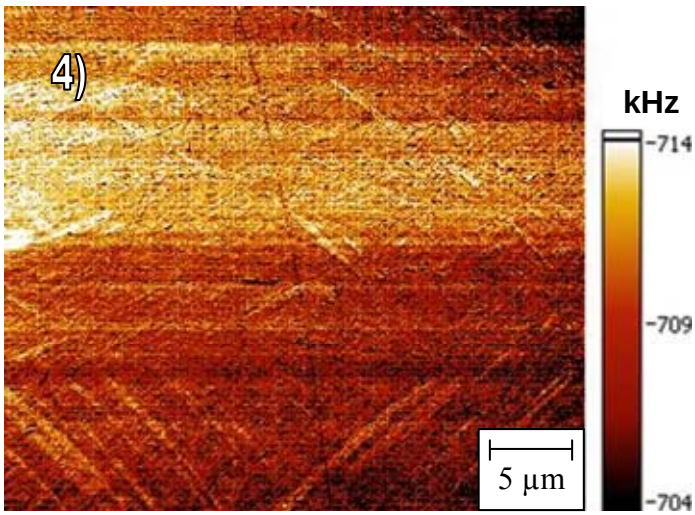
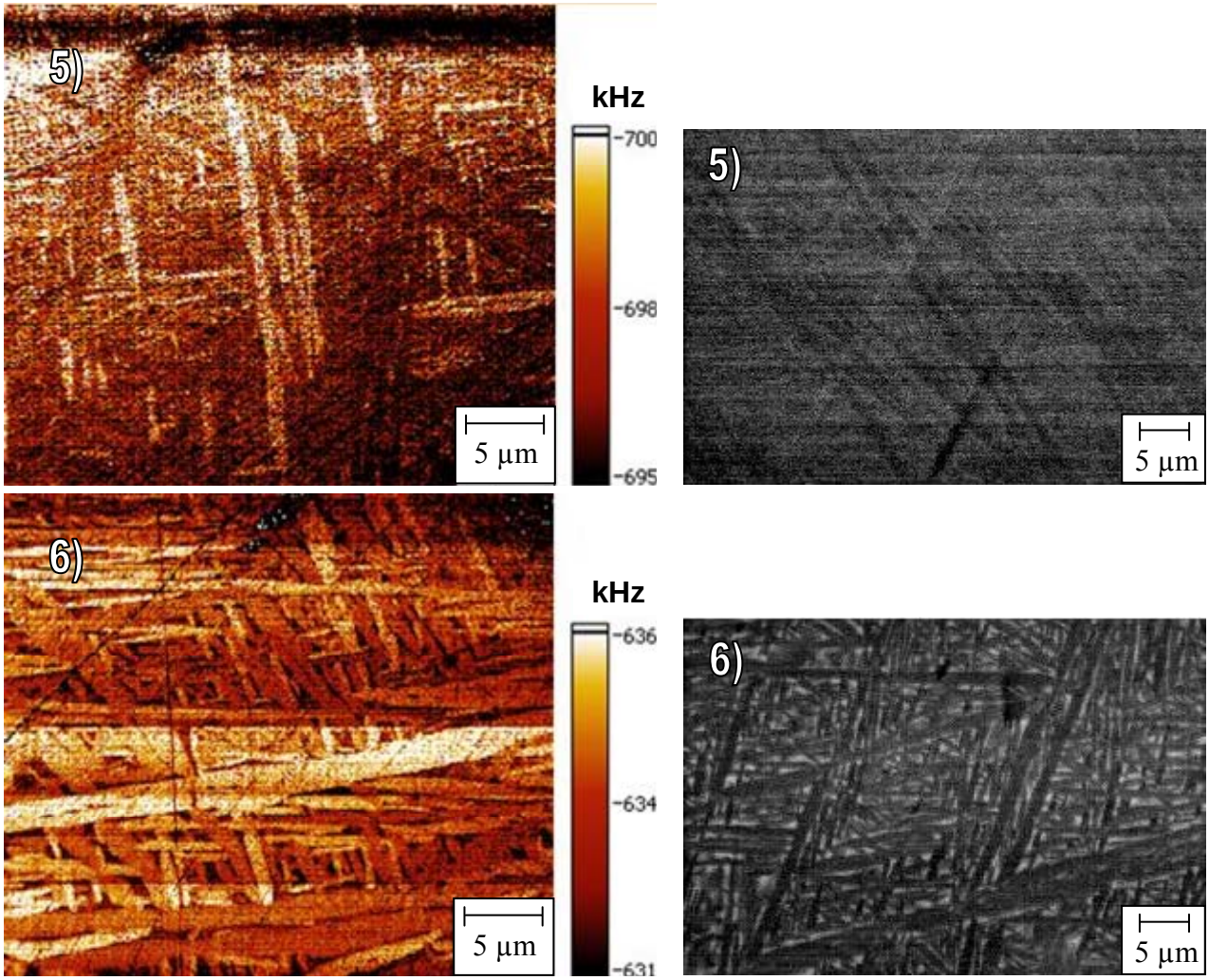


Figure 2. AFAM image of samples 4 after heat treatment at 1050°C (1h) followed by water quenching and without any further heat treatment. The 100% β-phase formed by the heat treatment at 1050°C transformed into 100% very fine needle-like α''-martensite structure, which is visible in the AFAM, but not in the BSE image.



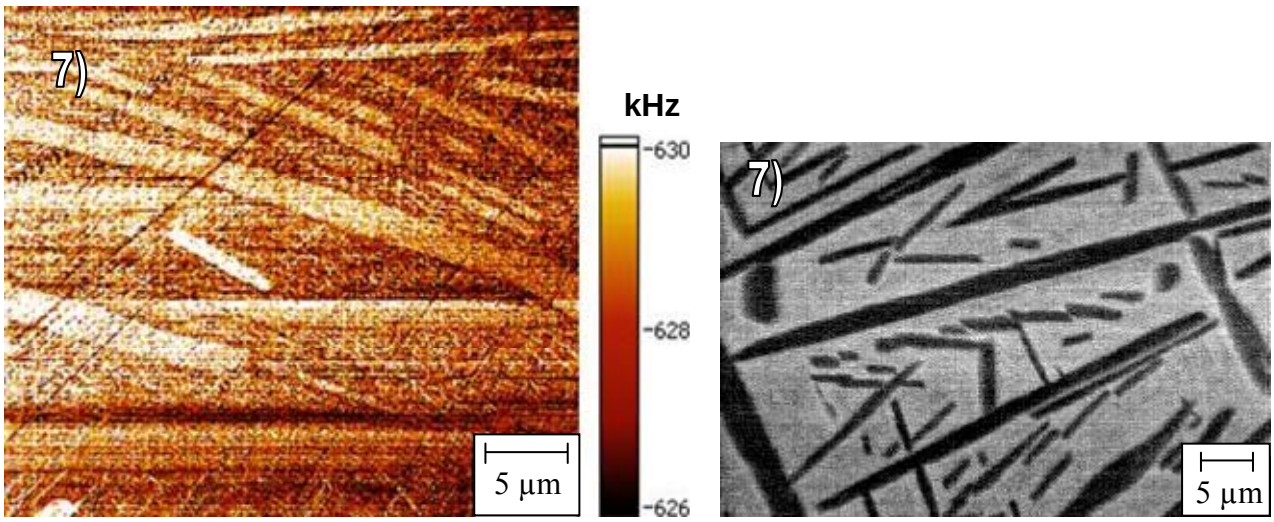


Figure 3. AFAM and BSE images of samples 5 to 7 after heat treatment at 1050°C (1h) followed by water quenching and a second heat treatment at 650°C, 850°C, and 950°C, respectively. In sample 5 long plates of α-phase were formed, the high contact resonance frequency indicates high stiffness. The β-phase formed between the plates shows lower stiffness. In sample 6 the stiff α-phase is thicker caused by the higher temperature applied and thus higher mobility of the atoms. Sample 7 was treated above the martensite transition temperature. Therefore, the β-phase has transformed into α''-martensite, which is of lower stiffness than the α-phase.

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