Microstructure and mechanical properties of annealed, quenched and suction cast Ti-6Al-4V

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Abstract. The effect of heat treatment or processing method on microstructure, mechanical properties and phase formation in Ti-6Al-4V alloys has been researched. The alloys in as-delivered, annealed, quenched and suction cast conditions were investigated using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and synchrotron X-ray diffraction (SXRD) analysis. The different processing- or heat treatment routes lead to significant changes in the phase composition. The quenched and suction cast alloys exhibited a mixture of martensitic α’ / α” and BCC β phase. In annealed and as-delivered alloys α’ + β phase mixtures was detected. The properties of alloys strongly depend on heat treatment and / or processing route.

1. Introduction
Ti-based alloys are currently one of the most attractive materials for application in medicine, since they are characterized by perfect corrosion resistance, high strength to weight ration and high biocompatibility [1, 2]. Since the properties of the alloys are depending on their phase composition, Ti-based alloys can be classified in three groups: α - alloys, α + β alloys and β alloys. The nowadays mostly used titanium alloys, Ti-6Al-4V is a typical example of a dual phase alloy. The martensitic Ti-6Al-4V alloy, which is suitable to both plastic working and heat treatment whereby its macrostructure and mechanical properties can widely be varied [3, 4]. The mechanical properties can be optimized by controlled adaptation of microstructure by thermo- or thermomechanical processing paths. For example the mechanical behavior of α + β and / or β Ti-based alloys can be significantly improved by a uniform distribution of fine α / α’ precipitations [5–10]. The transformation and precipitation pathways occurring during annealing decide the size, arrangement and morphology of the precipitation products.

In depth qualitative and quantitative studies of phase transformations and their influence on mechanical properties of the Ti6Al4V alloy are still lacking in literature, although better understanding of phase transformation kinetics during cooling from either α +β or β range seems essential. Therefore the main objective of the present work was to determine the effect of furnace cooling from 900°C (α +β range) on the mechanical properties and lattice parameters of Ti-6Al-4V.

2. Materials and Methods
The Ti-6Al-4V raw material used in this study was delivered in a form Ø 50 x 200 mm rolled bar. Small 20 x 20 x 10 mm³ specimens were cut from the bar normal to rotation axis. The Ti-6Al-4V_SC specimen was received by remelting and suction casting one of the above-mentioned specified specimen using a
BUHLER ArcMelter. For this, the specimen was placed in a triply flushed melting chamber, during melting an argon pressure of 800 mbar was maintained. For melting a water-cooled copper crucible and a non-consumable tungsten electrode were used. A titanium ingot was used as an oxygen getter and was melted prior to every melting procedure. The specimen was remelt 6 times and flipped after every melt. The received button shaped specimen was remelt and suction cast into a water-cooled cylindrical copper crucible with a diameter of 5 mm.

The Ti-6Al-4V_AN specimen was received by annealing the sample in a vacuum furnace at 900°C for 4h, followed by cooling in the furnace. The Ti-6Al-4V_Q specimen was heated to 1000°C, held for 0.5h and then cooled to room temperature by quenching in oil. For the Ti-6Al-4V_AD specimen no further heat treatment or other processing route was conducted.

The samples were mounted into phenolic resin and mechanically ground using SiC paper up to grade 1000 followed by pre-polishing with Al2O3 suspension. Final polishing was conducted using colloidal SiO2. The microstructure of the samples was revealed by etching with a Kroll’s reagent (5 vol. % HF, 10 vol. % HNO3 and 85 vol. % H2O). The microstructural analysis was performed using an optical microscope (Carl Zeiss AxioObersver Z1m). The hardness measurements, an average of 30 readings, were carried out using a WOLPERT Group 402 MVD Vickers hardness tester under a load of 50 g and a dwell time of 10 s. Synchrotron X-ray diffraction (SXRD) was conducted at the P07 beamline of the “Deutsches Elektronen Synchrotron” in Hamburg, Germany. Diffraction patterns were recorded by a 2-dimensional (2D) image plate detector with 2048 x 2048 pixels centered on the beam. An X-ray wavelength of λ = 0.124 Å and a beam cross section of 0.5 x 0.5 mm² were used. The sample-to-detector distance and diffraction center were calibrated at RT using a LaB6 powder. Azimuthal integration was carried out over the entire 360° to minimize potential effects of texture and grainsizes. The peaks in the diffraction patterns were fitted using Pearson-VII function and the peak parameters were calculated using least square method.

3. Results and Discussion

The SXRD patterns of quenched and annealed samples are plotted in figure 1. In both samples a mixture of $\alpha/\alpha' + \beta$ was detected. According to the higher intensities detected in the annealed sample it can be concluded that the volume ration of the $\beta$ phase is higher than in the quenched sample. Lower cooling rates typical for furnace cooling give rise to the transformation of $\beta \rightarrow \alpha$ or $\beta \rightarrow \alpha'$ from the high temperature $\beta$ phase. Considering that the initial Ti-6Al-4V samples consist at room temperature mostly of $\alpha$ phase (Al is a very strong $\alpha$ stabilizer) the weighted average lattice expansion of the two-phase mixture should be closer to that of $\alpha$ phase. When the $\beta \rightarrow \alpha$ takes place the ratio of $\beta$ to $\alpha$ increase and the expansion of the $\beta$ phase dominate. The lattice parameters of the samples are also shown in the figure. It can be seen that after annealing the lattice parameters of both phases increase, whereas the hexagonality of the $\alpha / \alpha'$ is nearly equal (1.5973 for quenched sample and 1.5974 for annealed sample).

Figure 2a shows the microstructure of the Ti-6Al-4V sample in as-delivered condition. A typical dual phase microstructure is visible. So called grain boundary $\alpha$ has formed along the boundaries. The suction cast sample showed in figure 2b shows $\alpha'$ being composed of long rectangular to each other oriented martensitic plates exhibiting an acicular morphology. The volume fraction of grain boundary $\alpha$ in the quenched sample, showed in figure 2c, was increased. According to the results of Ahmed and Rack this is a testimony of a reduced cooling rate [11].

Among all examined samples the Ti-6Al-4V_AD sample showed the lowest microhardness (388 HV0.05). The Ti-6Al-4V sample in as delivered condition is well known as $\alpha + \beta$ alloy. Both stable phases, $\alpha$ phase as well as $\beta$ phase are softer compared to their transformed phases like the martensitic $\alpha'$ or $\alpha''$ phases. The quenched and suction cast samples showed only a slightly different microhardness of 415 HV0.05 and 424 HV0.05. According to the SX-ray diffraction analysis showed in figure 1 a mixture of $\alpha' + \beta$ was obtained. Therefore, it can be concluded that the cooling rate in the suction casting process is as fast as during quenching. Both cooling rates are not high enough to force the formation of metastable $\alpha''$ phase in the samples. The highest microhardness is present in the annealed sample. This perceptible increase in microhardness was probably related to formation of metastable $\omega$ phase. It is assumed that
the cooling rate during the furnace cooling after annealing was small enough to subject the samples to an aging treatment.

![SXRD patterns](image)

**Figure 1.** SXRD patterns of a) quenched and b) annealed Ti-6Al-4V samples.

### 4. Conclusions

Based on the results obtained from characterization of the microstructure, phase composition and resulting mechanical properties, the following conclusions can be drawn. The microstructures of dual phase Ti-6Al-4V samples is sensitive to the heat treatment respectively the processing route. The cooling rate during the heat treatment or processing have a strong effect on mechanical properties of Ti-6Al-4V samples. The volume fraction of grain boundary α increased with decreasing cooling rate and the highest volume fraction was reached in the quenched sample. Among the studied alloys, the annealed sample showed a microhardness of 424 HV0.05 which is probably related to the presence of metastable ω phase.

### Acknowledgments

The work was carried out within the framework of the C-11 project of a competition among young scientists on the topic "Investigation of thermal stability and regularities in the formation of metastable phases of the Ti-Nb system for application in medicine" in 2018. Materials characterization was carried out at NSTU Materials Research Center.
Figure 2. Microstructure of Ti-6Al4V in a) as delivered, b) Suction cast, c) quenched condition and d) microhardness of the alloys

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