Influence of heat-treatment on Selective Laser Melting products – e.g. Ti6Al4V

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Abstract

Usually additive manufactured metal parts are showing a different mechanical behavior compared to conventionally produced parts used the same material. Apart from process-related macroscopic part imperfections (pores, surface roughness, etc.) the microstructure has a decisive influence on the mechanical properties of the materials. Thus, in order to optimize mechanical properties of metal parts a heat treatment for changing microstructures is routinely applied in most production lines to meet the product requirements. By means of the Titanium alloy Ti6Al4V the optimization of the static- and the fracture mechanical behavior by changing the microstructure with a heat treatment after the SLM process is discussed on the present work.

Introduction

The additive manufacturing (AM) details for metals has been published for about 20 years. First additive manufacturing processes were used primarily for rapid prototyping in variants of polymers and some metals [1]. Today, most of industry indicated their interest at manufacturing production by using selective laser melting (SLM) system work, rather than using AM for rapid prototyping. [2]. The mechanical behavior of SLM parts becomes vital information necessary to be used for component production.

It is important to accept the fact that the mechanical behavior of SLM parts are different to conventionally produced parts. Selective Laser Melting is a laser build-up welding technology with high cooling rates that result in a metal microstructure which is not comparable to metal casting or forged parts [7]. The dimension of difference from cast or forged parts varies depending upon the material being used. In all cases, the built SLM parts possess internal stresses, where the difference in mechanical behavior can be either an advantage or a drawback.

In most cases it is desirable to produce parts without internal stress. Currently, SLM parts employ a heat treatment for reduction of internal stresses but additional advantages are also possible from the heat treatment. The example of SLM parts out of Ti6Al4V will illustrate the mechanical property possibilities resulting from heat treatments. The primary aim of a heat treatment in this case of Ti6Al4V is the reduction of internal stresses. Due to the mechanical behavior of Ti6Al4V as build SLM parts, the heat treatment also seems to be essential to increase the mechanical behavior, e.g. the breaking elongation, the fatigue performance, etc.

Experimental Details

The investigation of material properties resulting from nine different types of heat treatments of Ti6Al4V SLM parts is presented within this body of work. The Ti6Al4V experimental tensile test bars and cantilever test specimens are built on a SLM250HL system from SLM Solutions GmbH in Germany with a 400W Ytterbium fiber laser. The Ti6Al4V powder particle size has an average value of 40µm. All used specimens were built using a 175W Laser Power setting and 30µm layer thickness. That is standard settings for laser melting of Ti6Al4V supported by SLM Solution GmbH.

The parameters for all nine tested heat treatments are shown in Table 1. The first variants of the heat treatments are performed at 750°C up to 850°C for 2h under an Argon gas atmosphere and cooled down inside the furnace. The next variants of the heat treatments are performed in vacuum with 950°C and 1050°C for 2h cooled down inside the furnace. Vacuum is used because the DMRC equipment is unsuitable for using Argon gas above a temperature of 850°C. Comparison tests between vacuum and Argon treated specimens show no important deviations. The last heat treatment variant was a hot isostatic pressing (HIP) process at 920°C for 2h.

Table 1: Parameters of heat treatment.

	1	2	3	4	5	6	7	8	9
Temperature / °C	20	750	775	800	825	850	950	1050	HIP
Time / h	-	2	2	2	2	2	2	2	2
Atmosphere	-	Argon	Argon	Argon	Argon	Argon	Vacuum	Vacuum	Argon

To illustrate the influence of heat treatment on *SLM-Ti64-parts* five experimental tests will be performed and explained within this paper; testing of internal stress with the help of cantilevers, testing of mechanical behavior with quasi-static tensile tests, HCF-tests or fracture tests and micrograph analysis.

The internal stress of SLM-parts cannot be measured at the DMRC so the deformation by cutting the cantilevers from the building platform is measured. The four cantilevers have a length of 44 mm and a thickness of 4mm (Figure 1). They are heated up in 25°C steps from 750°C to 850°C together with the building platform. A comparison with a non-heat-treated cantilever provides some indication of the development of internal stresses.

All quasi-static tensile tests are performed on a Universal Testing Machine Instron 5569 according to the ISO 6892-1:2009 standard [3]. The specimens (Form A20x4) are built with the gauge length in the z-direction and tested as build deviating from standard. A minimum of five specimens will be tested for each level of heat treatment temperature. The load will be measured with a 50 kN load cell and the elongation with an optical extensometer and with a measuring length of 20mm conducted at room temperature. The tensile stress R_m and the breaking elongation A_t are evaluated and presented within this paper.

A characterization of the microstructure of both the SLM-processed and the heat treated specimens provide information about microstructural behavior resulting from the heat treatment. A connection with the mechanical behavior and the microstructure is detected by an electron backscatter diffraction (EBSD) system. The EBSD scans are conducted at 20 kV on electro polished specimens with a scanning area of 30x30 µm with a step size of 0.15 µm. The scans will be differentiating between hexagonal grid structure (α',α) and body centered grid structure (β).

In addition to the quasi-static tensile tests, high cycle fatigue tests (HCF) are performed. The HCF-tests are run under the same atmosphere conditions as the quasi-static tests, at room temperature, and are performed on a Zwick/Roell Amsler HB250fatigue strength testing machine. The test runs are force-controlled with amplitude of 600 MPa and at a cylce frequency of 40 Hz. The mean force of the force cycle waveform is 0 N with a reversed force ratio R=-1. The round specimens are built in the z-direction and based on the standard ASTM E466 – 07.

The analyses of the crack growth behavior and fracture tests are run on an Instron testing machine ElectroPlus™ under room temperature (20°C). The test specimens are manufactured according to the standard of ASTM E647-08 and were built in the z-direction. The tests are run with a force ratio of R=0.1 and in the area of K-decreasing (area of crack initiation) with lower crack growth rates by a frequency of 40 Hz and in the area with higher crack growth rates by a frequency of 10 Hz.

Results

The first aim of heat treatment is for the reduction of internal stresses. Strain measurements after cutting the support structure of the cantilevers are plotted in Figure 1. Figure 1 shows a comparison of the non-heat-treated specimen (20°C) where there is no considerable bending measured. The relation between internal stresses and the bending of cantilevers is a good index for measuring the reduction of internal stress. For all the tested temperature levels from 750°C to 850°C, the measurable strain reduces from 0.32mm to 0.05mm or less.

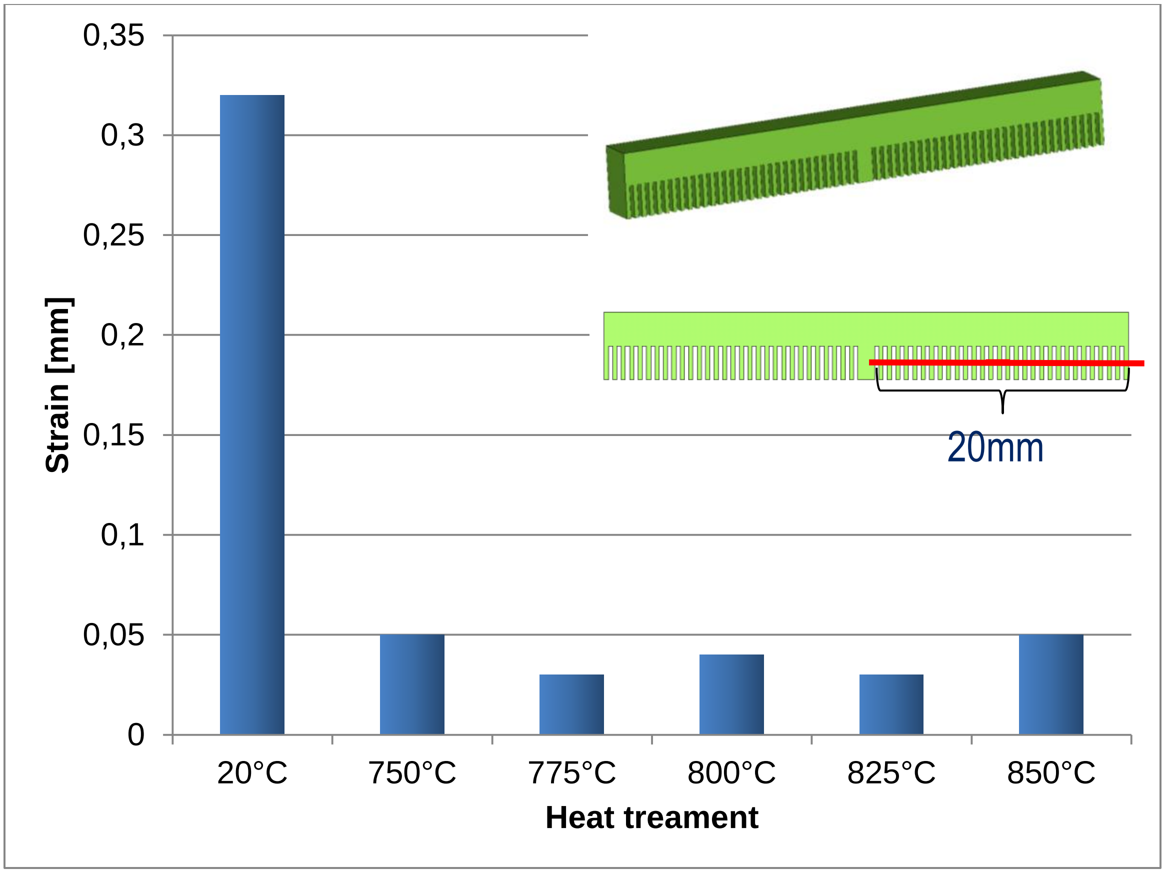

Figure 1: Bending of Ti64 cantilevers after heat treatment.

The results of tensile tests are shown in Figure 2 and Figure 3. All results are produced with specimens that were tested as built in the z-direction and compared non-heat-treated specimens. Figure 2 shows the measured tensile strength R_m and Figure 3 shows the appropriate breaking elongation A_t. The first bar in both diagrams representing the specimen without any post process shows a nearly brittle structure. Due to that, while the decreasing of tensile strength from 1080 MPa to 945 MPa seems to be fractional (12.5%) the increasing of breaking elongation from 1.6% to 11.6% (625%) is reached. For example international standards for *implants for surgery* are claiming special minimum values for the tensile strength and the breaking elongation especially for Ti6Al4V [4]. Therefor the tensile strength should realize a minimum of 860 MPa and the breaking elongation a minimum of 10%. In case of SLM produce parts this requirements are fulfilled by heat-treatment.

The reason of such an increasing of breaking elongation can't be described with the reduction of internal stress shown in Figure 1. Internal stress isn't already detectable after a 750°C heat treatment but also with an 850°C heat-treatment a maximum of only 5.2% breaking elongation in average can be reached. Due to this, other effects have to explain the optimization of breaking elongation up to 11.6% in average and a view to micrographs seems to be necessary.

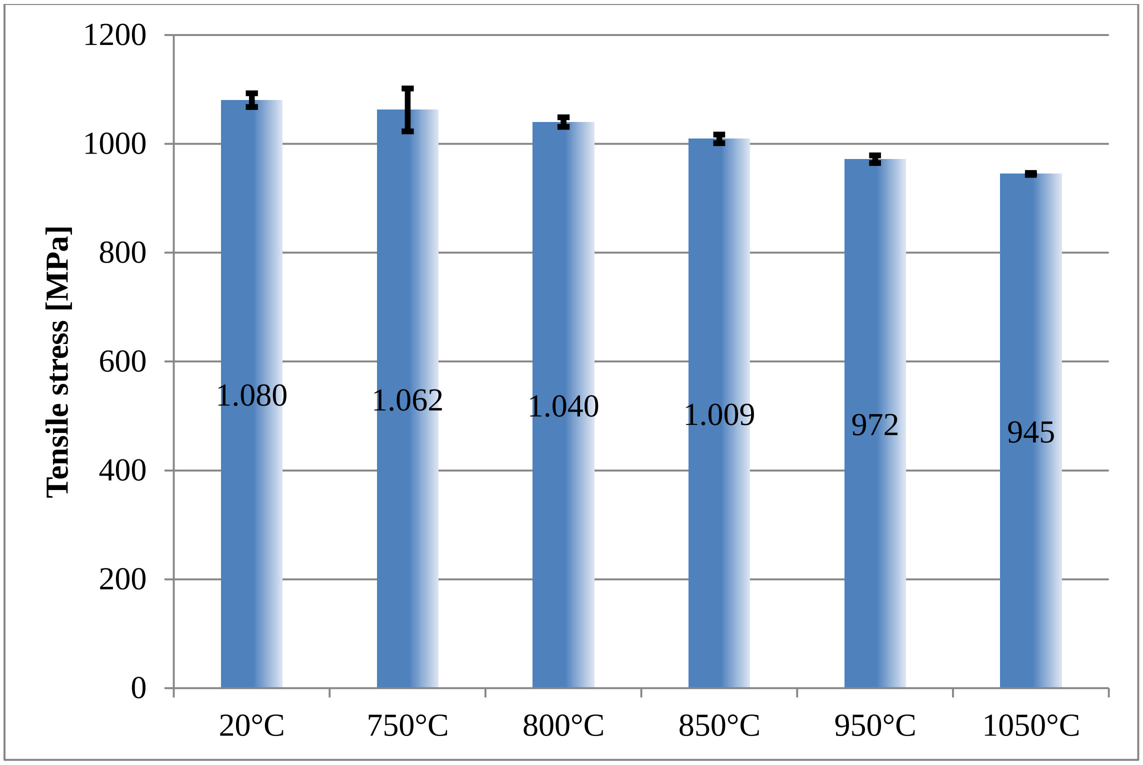

Figure 2: Tensile stress of heat-treated specimens

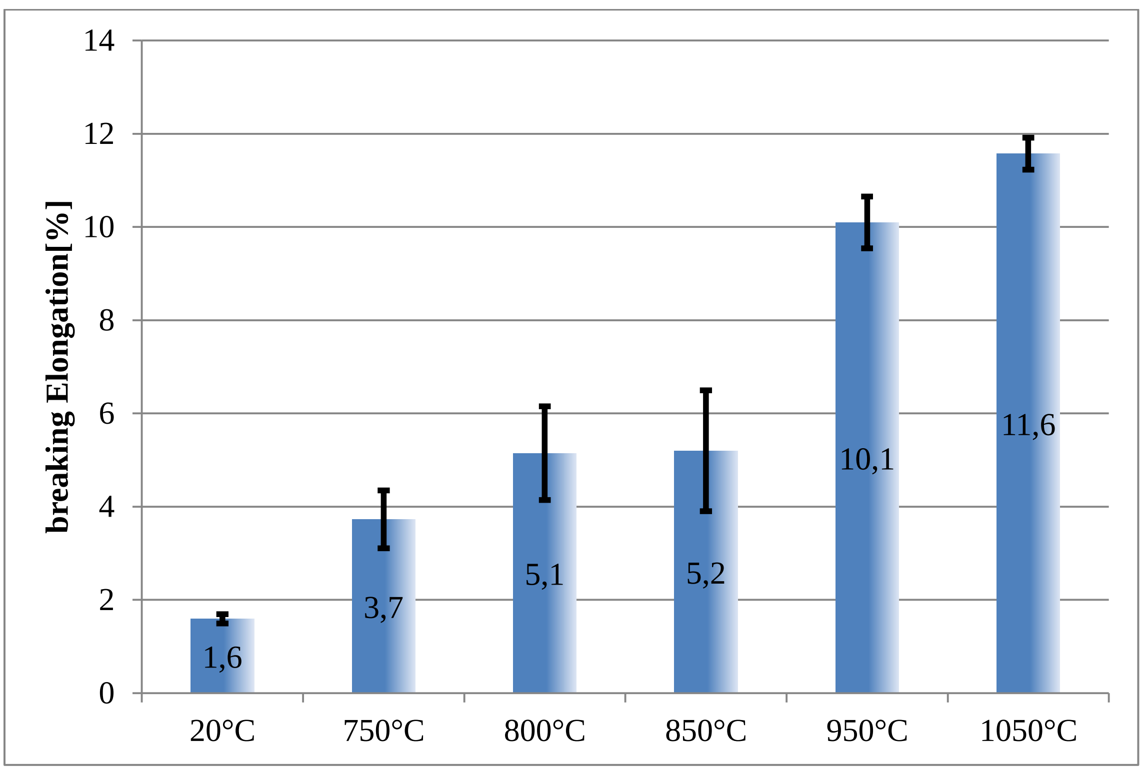

Figure 3: Breaking elongation of heat-treated specimens

In addition to tensile testing, looking at the microstructure to determine the phase development of α and β titanium is also investigated. A SEM is used to detect structural elements and identify them as α and β constellations with the help of an electron back scatter diffraction (EBSD) scan. Figure 4 shows two EBSD micrographs of a Ti6Al4V specimen as build (left) is compared with a 950°C heat treated specimen. First notice that the laminar structures of the non-heat-treated specimen (left side) are very fine when compared to the 950°C heat-treated specimen (right side). That is one reason for an increasing of breaking elongation because sliding effects are mainly detected between grains [5]. The second effect can be illustrated with the help of EBSD scan and the following determination of hexagonal grid structures (α', α) and body centered grid structures (β). The difference between the martensitic α'-phase and the α-phase is only a minimal structure distortion and is not detectable with EBSD. Due to the high cooling rate inside the SLM process the probability of formation of an α'-phase in the non-heat-treated specimen is very high [6,7,8]. The α-phase and the α'-phase are colored in red where the body centered β-phase is colored in green which were only detected in the heat-treated specimens. The β-phase is mainly detected on the edge of laminar structure and also supported a more ductility behavior of the material [5].

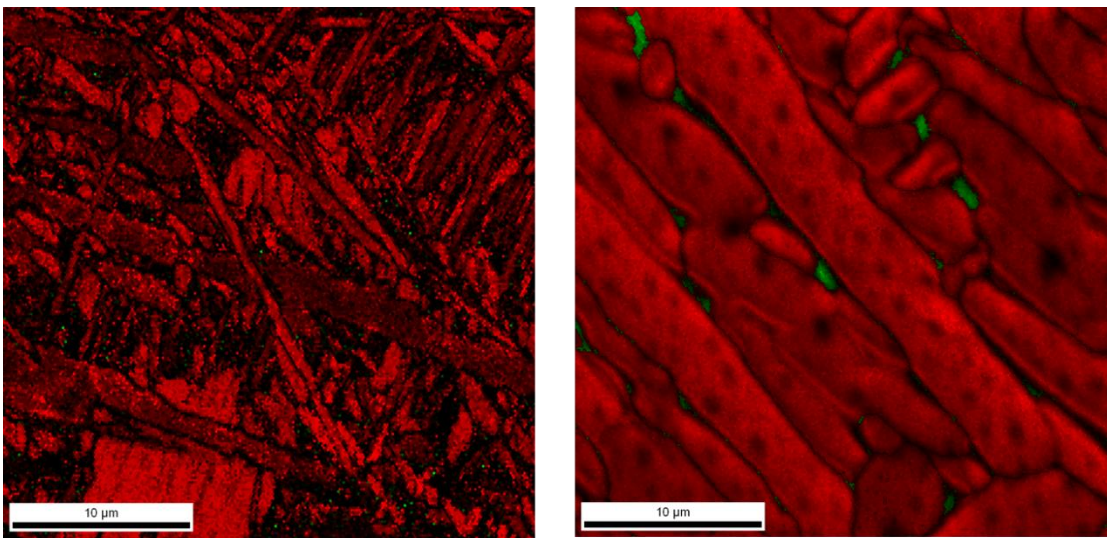

Figure 4: Comparison of EBSD scans (30µm x 30µm) between non-heated (left) and after the 950°C heat treated specimens (right)[5].

In addition to the analysis of quasi-static behavior it is of interest to take a look at high cycle fatigue behavior (HCF). Table 2 list the results of HCF tests described in the experimental details. Table 2 shows the different heat treatments, the frequency of cycles, the stress level and the endurance of fatigue life N_f, together with the standard deviation. For all treatments a minimum of 5 specimens are tested [9]. .

Table 2: Results of high cycle fatigue tests

Treatment	Frequency/Hz	Stress/MPa	Cycles	STDV
20°C	40	600	28,900	±11,000
800°C	40	600	93,000	±16,800
1050°C	40	600	290,000	±76,000
HIP	40	600	>2,000,000	

The first three heat-treatments are shown before by the quasi-static tests (Figure 2 and Figure 3). The number of cycles to failure increased with the increasing of temperature heat-treatment. That improvement is similar to what was observed in the quasi-static test results. By changing the microstructure 290,000 cycles are reached. But in no single case it will be reached a level of 2,000,000 cycles to failure. Pores are mostly the initiators of crack growth and result finally in failure of complied parts. Especially pores close to the edge proves to be bad because these pores affect the stress distribution on a micro level [10]. Besides the changing of microstructure it seem to be very important to minimize the number of pores especially at areas close to the edge. To close most of the pores in a post-treatment process in the last case of Table 2 hot isostatic pressing is used. A combination between a heat treatment for changing the microstructure and a pressure treatment for closing pores.

Also for crack growth curves like shown in Figure 5 with the combination of a heat treatment and a pressure treatment, the same characteristics of the parent material from Ti6Al4V can be reached [9].

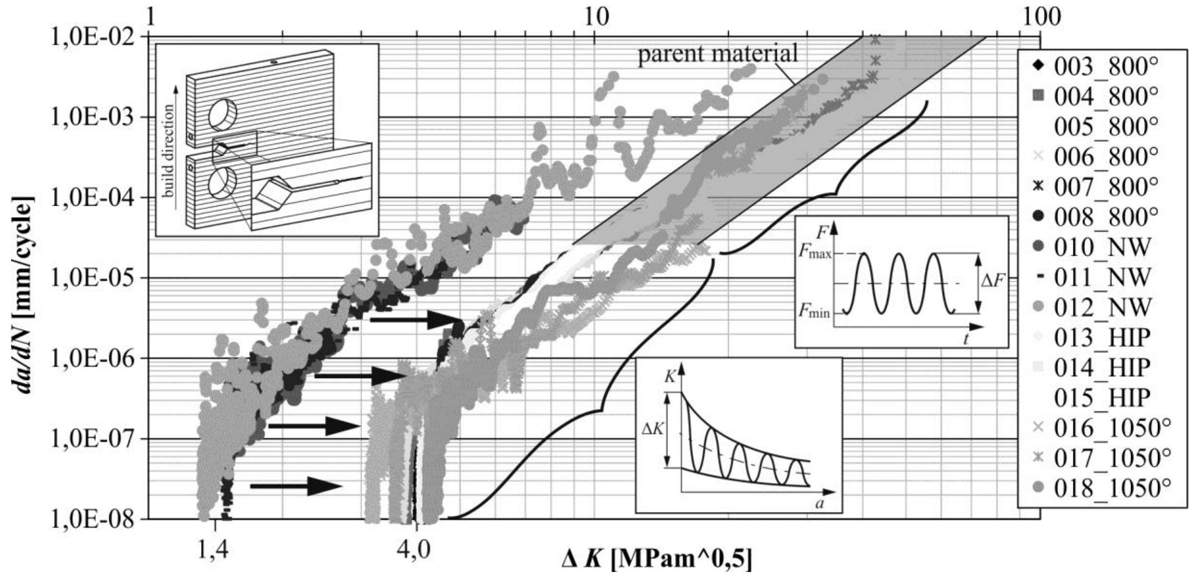

Figure 5: Position of crack growth curves non-treated and heat-treated compared with parent material [9]

Conclusions

A close investigation of the mechanical behavior of Ti6Al4V and an evaluation on the influence of heat treatment of SLM-products is given. Due to changes of microstructures three cases of treatment have been identified (Figure 6). In most cases the SLM parts as built possess internal stresses and have a very small sized microstructure. In case 1 (Figure 6) using a heat-treatment is used in the reduction of internal stress without any changing of microstructure. By reduction of internal stress a first beneficial effect is detectable regarding to the mechanical behavior e.g. the increase of elongation at break. By decreasing the internal stress for conservation the shape of SLM parts during cutting parts form the build platform is also a main topic of additive manufacturing. This case 1 of heat treatment can be mostly realized with a short heat-treatment on a low temperature level. Case 2 in Figure 6 is the changing of microstructure of SLM parts by using a higher temperature level or a longer annealing time for heat treatment. The meaning of changing the microstructure is to enlarge the grain size or the laminar structure (e.g. Ti64) and to change the constellation of microstructure, e.g. from a α'-constellation to a αβ-constellation of Ti6Al4V. And case 3 in Figure 6 is the changing of microstructure combining with reduction of pores by using hot isostatic pressing. In addition to showed optimization of quasi-static behavior in this case also the fatigue properties optimized by closing especially surface near pores.

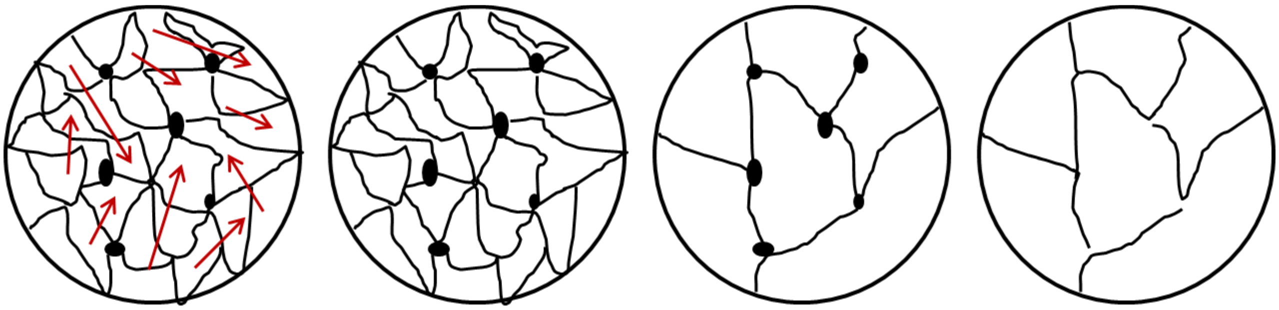

Figure 6: Three cases of microstructure manipulation

Acknowledgments

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