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Study on Thermomechanical Treatment, Mechanical Properties and Fatigue of Nitinol Superelastic Thin Sheet

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A wide range of different thermomechanical treatments was performed on commercially available superelastic Nitinol thin sheet. The ingot composition in the range of standard superelastic material with about 50.8 at.% Ni, balance Ti, was used to manufacture a series of samples with different thermomechanical conditions. Production parameters such as cold work, heat treatment temperature, and heat treatment time were varied. All finished samples were of the same final thickness of 0.3 mm and received the same industrial surface finishing process to obtain a smooth, defect, and oxide-free, shiny surface. Before carrying out the laser cutting, the material was characterized by tensile testing, DSC, and bend-and-free recovery test. Miniature dogbone specimens were cut from the as-manufactured sheets in both directions, longitudinal as well as transverse to the rolling direction. These samples were surface finished using standard deburring and electropolishing processes. For some specific parameter combinations, there were also samples taken at 45° to the rolling direction. All qualified samples were then exposed to fatigue testing in a bending mode until fracture or run-out. The results showed there is a significant effect on the fatigue performance of the samples from both the applied thermomechanical treatment as well as the sheet anisotropy. It is also obvious that the achieved strain data is on average lower than the data obtained in comparable studies on tube or wire, which can be attributed to the different test setup (bending mode in air at 37 °C) as compared to most other studies as well as the larger surface.

Keywords heat treating, mechanical testing, nonferrous metals, rolling

1. Introduction

Nitinol superelastic sheet has become very popular over the last couple of years, which is attributed to improvements in the material supply chain and the fact that many engineers have realized the additional design opportunities of Nitinol sheet. Furthermore, sheet-based products offer to medical OEMs the opportunity to differentiate their designs from their competitor's products. Lastly, sheet material offers unique and different processing opportunities based on punching, photoetching, or deep drawing processes, all of which are not available for Nitinol tube.

Contrary to the undisputed success of Nitinol tube when it became available for stent manufacturing, the market penetration of Nitinol thin sheet appears to be slower, mainly because the uniquely achievable configurations of sheet-based products are not as obvious as for Nitinol superelastic tube. However, over the last couple of years the market for thin sheet has grown continuously and the overall annual material consumption is estimated to be approximately 4.000 kg. This is mainly driven by volume applications, such as

- Abbott Vascular StarClose™ (closure device for healing the access wound after catheter-based procedures).
- 3M Unitek SmartClip™ (clip function of a self-ligating bracket).

Besides many others, these applications consume millions of parts per year and have positively affected the sheet material consumption over the last couple of years. Furthermore, a few applications in the nonmedical field—most of them related to the high damping capability of superelastic Nitinol—have surfaced and are contributing to the material consumption in this area with as much as 20%.

Relatively few papers have been published about work on the thermomechanical treatment of commercial Nitinol sheet (Ref 1-3) and the resulting mechanical and thermal properties in different sheet orientations in relation to the rolling direction.

The purpose of this article is to characterize the mechanical and thermal properties of commercial superelastic Nitinol sheet after different sets of applied production parameters. Special emphasis is put on the dependence between mechanical properties and sample orientation. Furthermore, the fatigue performance in bending strain conditions is investigated.

2. Experimental

Nitinol thin sheet with an ingot composition in the range of standard superelastic material with about 50, 8 at.% Ni, balance

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Ti was used to manufacture a series of samples with different thermomechanical conditions. The samples were produced by initial hot rolling to a final thickness of about 2 mm and subsequent cold rolling with thickness reductions between 30 and 40%. Interpass anneals around 650 °C were carried out to relieve the hardening from the cold work. The final process parameters for the final reduction steps were tightly controlled. Production parameters such as cold work, heat treatment temperature, and heat treatment time were varied. The list of parameters used for this study is summarized in Table 1

The final heat treatment process was conducted in a special custom-built machine with heated tool plates while intentionally applying rather short heat treatment durations to simulate industrial processes which are usually based on shorter furnace residence times. The heat treatment time was varied between 3 and 10 min. Instead of increasing the annealing time between the heated tool plates, it was decided to take the samples into a subsequent air convection furnace heat treatment at the same temperature as the tool plates. The combined heat treatments were utilized as the heated tool plates were not designed for heat treatment times longer than 5 min. Furthermore, the long heat treatments would have extensively consumed the equipment which is heavily used for regular sheet manufacturing. Thus, the heat treatments carried out at 10 and 30 min were to be considered two-step heat treatments at the same temperature. The cooling of the sheet coupons was carried out on a water-cooled steel plate surface as water quenching of hot flat annealed sheet coupons tends to initiate internal stresses which lead to lack of flatness after quenching.

All finished samples were of the same final thickness of 0.3 mm and received the same surface finishing process to obtain a smooth, defect, and oxide-free, shiny surface (chemical oxide removal). Before laser cutting, the material was characterized by tensile testing (ASTM F-2516), DSC (ASTM F-2004), and bend-and-free recovery (BFR, ASTM F-2082) test. Afterward, miniature dogbone specimens according to Fig. 1 were cut from the as-manufactured sheets in both directions, longitudinal (rolling direction, RD) as well as transverse to the rolling direction (transverse direction, TD). These samples were surface finished using a standard deburring and electropolishing process. On a few sheet coupons, samples were also taken at 45° orientation to the rolling direction.

All qualified samples were then exposed to fatigue testing similar to a 3-point bending mode until fracture or run-out (criteria for run-out: minimum 5 million cycles). The testing was performed at a frequency of 60 Hz in 37 °C constant air temperature conditions. The fatigue tester was custom-built and is also used on regular production work orders (Fig. 2). As opposed to the free-free boundary conditions which are typically used for a 3-point bending test, the setup used for this study applied fixed-free boundary conditions, but allowed both arms of the samples to rotate without any constraint (see detail in Fig. 2b). This was achieved by means of support points made from small steel balls. During unloading, the sample is fully unloaded to the zero stress condition. The applied stress is not reversed by the test machine. The strain in the sample was approximated using a linear elastic strain calculation model as described in Ref 4.

Not all possible combinations of thermomechanical treatments have shown good tensile properties due to lack of superelasticity. These samples were not qualified and therefore not taken into consideration for further testing. Thus, the

Table 1 Summary of the process parameters applied during the manufacturing of the sheet samples

Cold work, %	Annealing temperature, °C	Annealing time, s	Sample orientation, °
25/30/35	480/495/510/525/540	180/600	0/90/45

Not all combinatorial possibilities were measured. The samples annealed for $t = 600$ s were exposed to heat treatments in two steps: step 1 for 180 s in a heated plate tool and additionally for a second heat treatment in an air convection furnace. All heat treatments were followed by natural convection cooling of the sample material on a cold steel plate

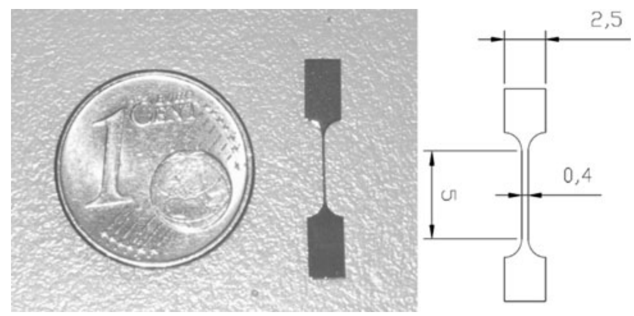


Fig. 1 Sample geometry of the as-laser cut dogbone (left-hand side) and the drawing with dimensions (right-hand side)

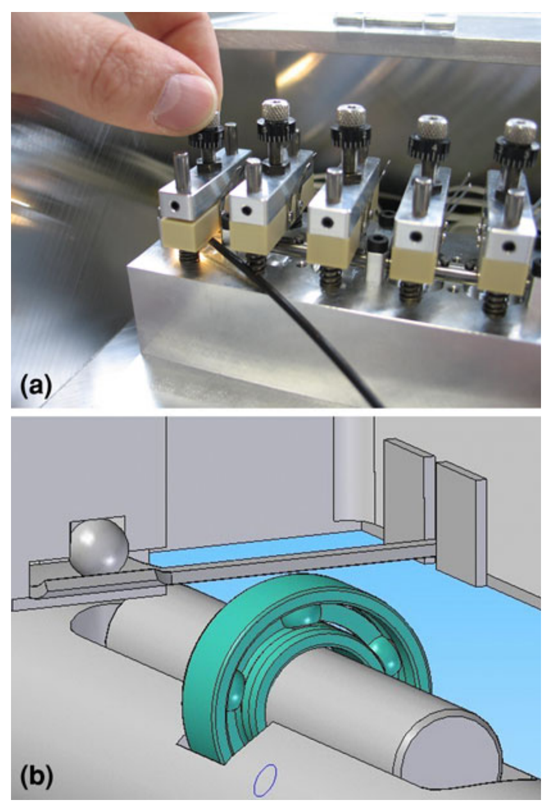


Fig. 2 Sample setup in Admedes bending fatigue tester (overview in Fig. 2a, detail about the sample holder in Fig. 2b). The testing is carried out in moved air ($T = 37$ °C) with a frequency of about 60 Hz. As many as 10 samples at a time can be tested

number of reported samples was reduced accordingly during the course of the program.

3. Results and Discussion

3.1 DSC and BFR Testing

All produced sheet materials were measured in a DSC using an autosampler to determine the transformation temperatures and transformation enthalpy. The results are summarized in Fig. 3.

It is obvious that the transformation temperatures decrease with increasing heat treatment temperature. The differences for the measured A_f values between 25, 30, and 35% cold work are not significant. This is not in full correlation to the existing published data on wire and tube, especially for the lower annealing temperatures (Ref 5, 6). Here, one should expect decreasing transformation temperatures with increasing amounts of cold work. In wire and tube, the increasing amount of dislocations with increasing cold work leads to a decrease of transformation temperatures. This general finding was not confirmed with sheet production. The reason for this will be the subject for future research work.

Another result from the DSC and BFR measurements is shown in Fig. 4. For lower heat treatment temperatures, the correlation between DSC and BFR becomes worse. This is most likely resulting from the stabilization of the R-phase with lower heat treatment values. It can be concluded that the BFR measurement tends to be more affected by stabilization of the R-phase, while this is certainly not the case for DSC results where the peaks in most cases could be clearly differentiated.

3.2 Tensile Testing

During tensile testing of the samples it became obvious that there were no significant improvements achieved by longer heat treatments. Most of the tensile curves obtained showed less than full superelasticity, with very low plateau stresses and incomplete transformations during unloading. Thus, it was decided to discontinue the work on such samples with heat treatment durations longer than 3 min. This also reflects the standard industrial process of flat annealing Nitinol sheet stock, which is based on rather short heat treatments. The variation of the heat treatment duration was taken off the research program during the course of the work.

The results showed the following parameters to have the most significant effect on the tensile properties:

- amount of applied cold work
- sample orientation.

Figure 5 shows the effect on UTS and upper plateau strength (UPS) as a function of cold work and heat treatment temperature. The reported heat treatment time was always 3 min. A correction of differences between test temperature (37 ± 2 °C) and active A_f temperature was not applied for the

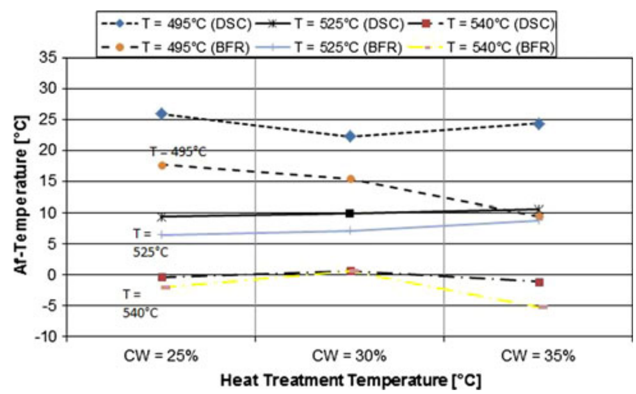


Fig. 4 The effect of cold work on the measured A_f values and comparison between DSC and BFR data. It becomes visible that the cold work does not affect the transformation temperatures in a similar way as found in wire and tube. Also, the correlation between DSC and BFR becomes worse with decreasing heat treatment temperature, which may be attributed to the increasing R-phase peak that has a potentially stronger effect on the BFR test results

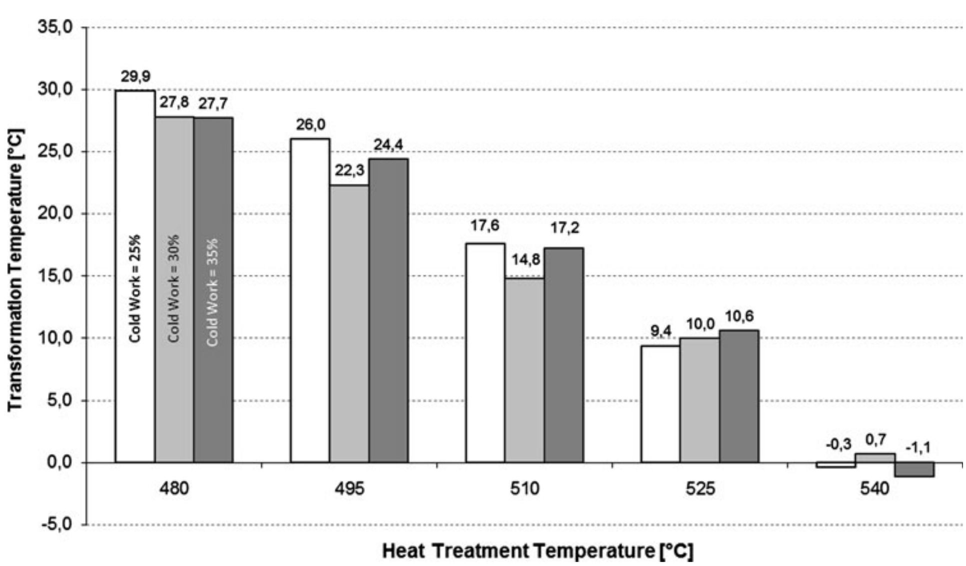
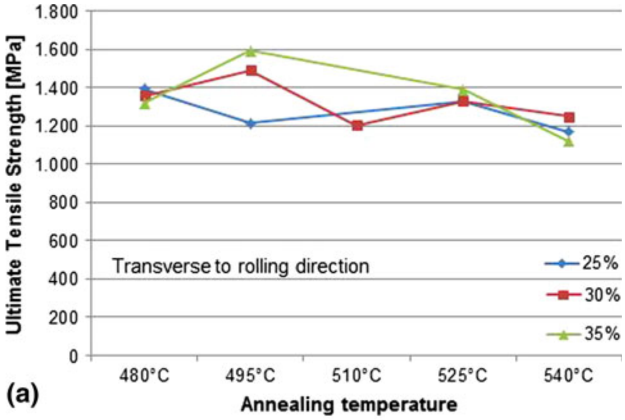


Fig. 3 Results of DSC measurements after thermomechanical treatment (A_f value). The basic results are in line with data collected from wire or tube measurements



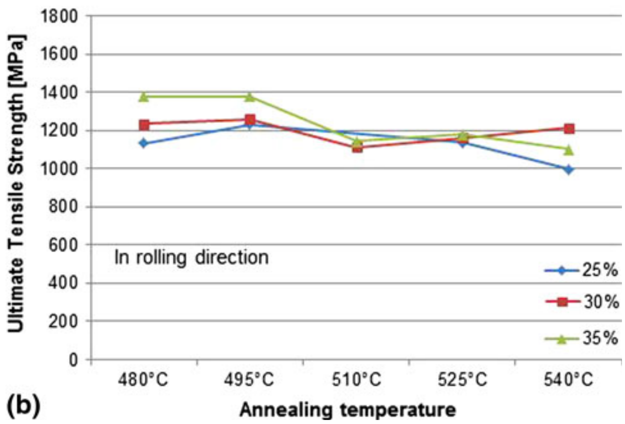


Fig. 5 Results from tensile testing (UTS) in transverse (upper graph) and parallel to the rolling direction (lower graph). The differences between the applied cold work amounts are smaller after higher heat treatment temperatures. Higher heat treatment temperatures lead to lower UTS values. The results obtained from samples in rolling direction are in general lower than those from samples transverse to the rolling direction

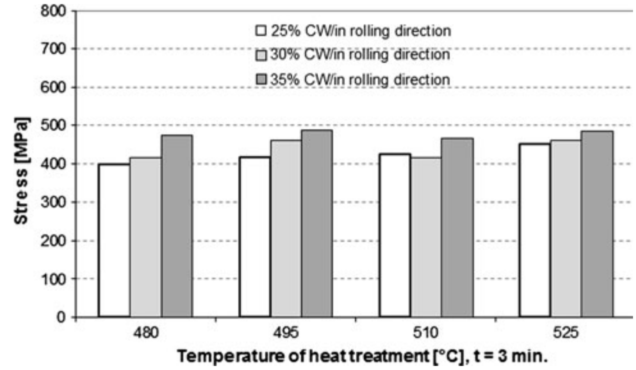


Fig. 6 The effect of cold work on the measured plateau height at a total strain of 3% (loading plateau). The plateau height is mainly affected by the amount of cold work, whereas the heat treatment temperature is less effective in controlling the plateau stress

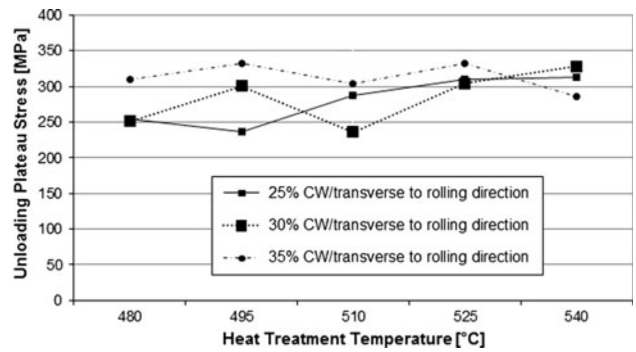


Fig. 7 The effect of cold work on the measured plateau height at a total strain of 2.5% (unloading plateau, test temperature 37 °C). The plateau height is hardly affected by the heat treatment temperature. An increasing amount of cold work leads to higher values for the unloading plateau. Furthermore, the measured unloading plateaus are higher if compared to samples taken in rolling direction

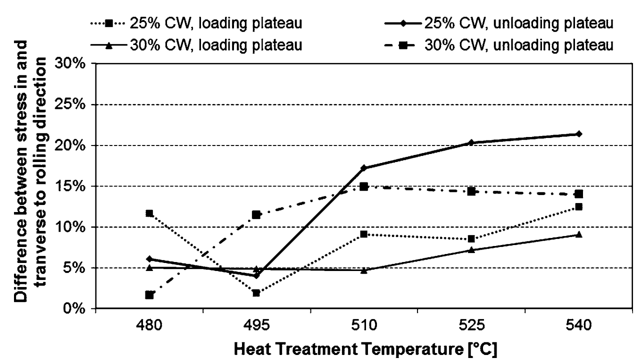


Fig. 8 The effect of heat treatment temperature and cold work on the plateau height at a total strain of 3.0% (loading plateau, test temperature 37 °C) and at 2.5% (unloading plateau, test temperature 37 °C). The height of the plateaus in rolling direction is always lower as compared to the plateaus from samples transverse to the rolling direction. The difference in height increases overall with increasing heat treatment temperature

tensile tests. A general trend toward lower strengths with increasing annealing temperatures was observed as expected.

For increasing annealing temperatures, the difference between samples with lower and with higher amounts of cold work is reduced. The effect of cold work on the measured UTS values is rather small in rolling direction and tends to be higher in transverse direction.

The UTS in rolling direction was significantly lower than the UTS in transverse direction (between 5 and 25%). The same statement holds true for all other mechanical data, such as the UPS and the LPS. If we look at the difference between longitudinal and transverse samples (Fig. 6 to 8), we also find a consistently higher value for the plateau stress of the transverse samples.

This effect can be attributed to the higher amount of grain and subgrain boundaries in samples transverse to the rolling direction. Even though this has not been verified during this study by means of SEM/TEM, the conclusion is very likely and in line with general expectations.

Another interesting result of this study is shown in Fig. 9.

The samples with a 45° orientation show the highest plateau stresses of all measured samples which is consistent with findings from other authors (Ref 1, 2). Unfortunately, this phenomenon cannot be explained by the qualitative approach followed in this study and will also need further research work to really understand the nature of this effect. According to Lin and Boylan (Ref 1) the root cause might be in the increased availability of preferred crystal orientations (such as $[111]_{B2}$ or

$[011]_{B2}$) in rolling direction, while the transverse direction shows fewer suitable variants with high calculated transformation strains. Hence, the stress-induced phase transformation may occur at a higher stress level in transverse direction.

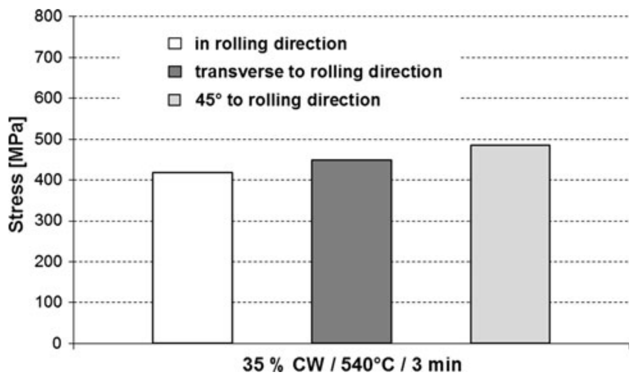


Fig. 9 The effect of the rolling direction on the height of the loading plateau. Interestingly, the samples taken in 45° orientation show the highest loading plateau stresses

4. Fatigue Testing

The accelerated fatigue test using Mini-Dogbones delivered the results shown exemplary in Fig. 10. This figure shows results for different amounts of cold work after 480 °C/3 min and after 540 °C/3 min in rolling as well as in transverse direction. There is a general tendency toward a decreasing strain with increasing number of cycles. From the measured data there seems to be no endurance limit for higher number of cycles.

The measured differences between the sample families are not very big and the findings do not suggest any major effect of the sample orientation. It appears that an increasing amount of cold work supports longer fatigue life and that the transverse direction also is advantageous if looking at higher cold work.

The measured data is not consistent with literature data that was mainly recorded for Nitinol thin wire in rotating beam testing (RBT) (Ref 7). However, for a higher number of cycles the results in Ref 7, 8 are in some respect in correlation with the findings from this article, as the differences between the different amounts of cold work are quite small in the high cycle fatigue (HCF) range.

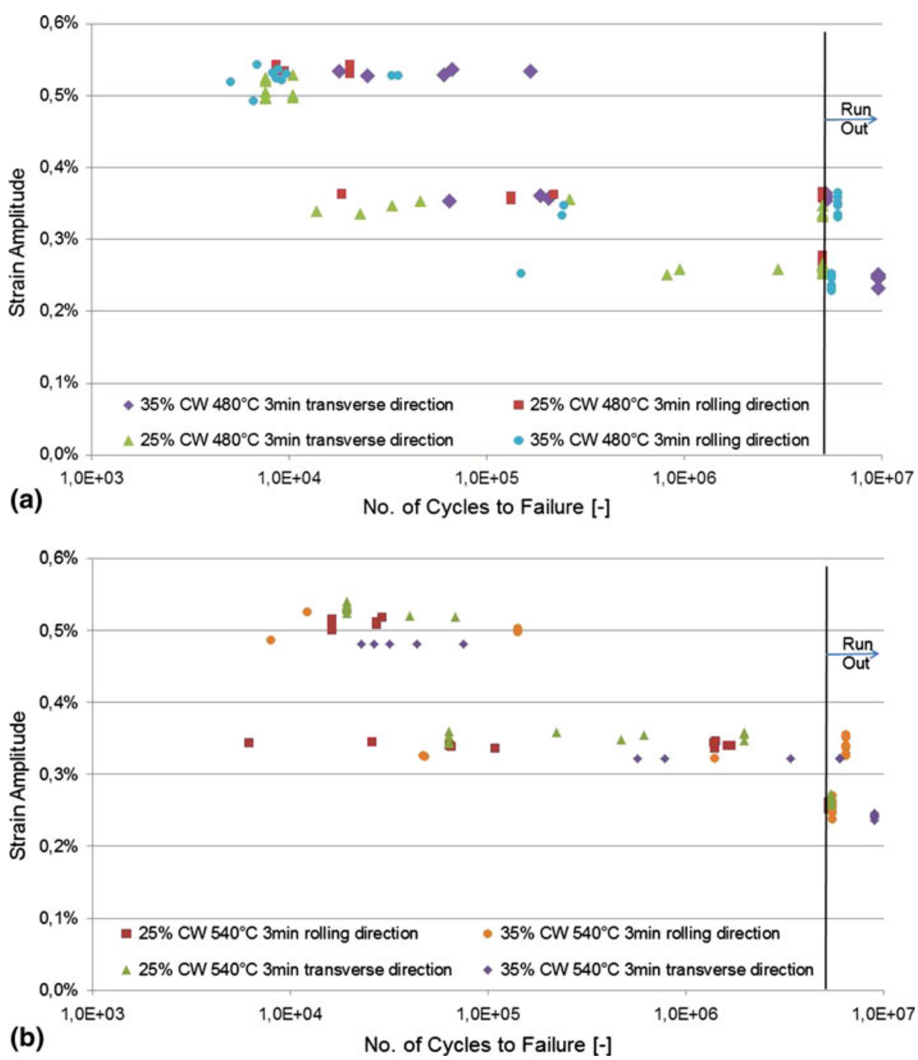


Fig. 10 Effect of thermomechanical treatment and sample orientation on the fatigue behavior of the test dogbone samples. All data points on the right side of the run-out line (5 Mio. cycles) did not break and the testing stopped at the shown number of cycles (run-outs). Upper: heat treatment at 480 °C/3 min, lower: 540 °C/3 min. There appears to be a tendency toward higher strain at a given N for higher cold work (35%), even though the differences are not significant

One aspect in the current test setup needs to be considered for future testing: the temperature control of the sample test chamber is quite limited as the air temperature is controlled, but the sample temperature may be quite different due to the relatively high frequency of 60 Hz. This effect was partially offset using a very thin cross section of the samples (see Fig. 1), but the limited control of the sample temperature during fatigue testing remains a fact and is still a concern. This may lead to early fractures if the samples heat up during testing. Another difference between the test setups is in the RBT versus the 3-point bending test that was used in this article. Other authors also reported much lower endurance limits if they applied different test methods such as tension-tension on wire (Ref 9). To our understanding it only allows a qualitative comparison of the results achieved within this study with any of the articles using rotating beam on wire for fatigue testing.

5. Conclusions

The results show that the expected sheet anisotropy significantly affects the mechanical performance of the thermomechanically treated samples. The average UTS and plateau stresses are consistently higher in transverse as compared to samples taken in the rolling direction. This was attributed to texture effects (average grain size expected to be smaller in transverse direction), but is mainly caused by formation of preferred martensite variants for samples in rolling direction.

The measured DSC and BFR data are in good correlation to the results from wire tested in similar studies from other authors.

During the fatigue testing it became obvious that the achieved strain data is on average somewhat lower than the data obtained in comparable studies using RBT on tube or wire. This can be attributed to the accelerated fatigue test setup (3-point bending in air) as compared to most other studies as well as the larger surface and the more complex geometry and deformation situation of the samples. The differences between transversal and longitudinal samples were insignificant, whereas higher amounts of cold work slightly increased the amount of strain for a given number of life cycles to failure. Best results were obtained for average amounts of cold work (30%) after heat treatments in the 510 to 525 °C range.

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