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To cite this article: Harm Kooiker et al 2018 J. Phys.: Conf. Ser. 1063 012106

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The evolution of mechanical properties of AISI 301 as a result of phase reversion heat treatment, experiment and modeling

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Abstract. Laser heat treatments of metastable austenitic stainless steel AISI 301 are presented aiming to elucidate the relation between heat treatment, transformation and mechanical properties after heat treatment. It is assumed that the observed phase reversion of martensite to austenite is due to a diffusional transformation mechanism governed by nucleation and growth leading to submicron grains. Based on this assumption it is demonstrated that the reverse transformation can be successfully predicted by the proposed model. Subsequently the effect of the heat treatment on the hardness is reviewed. It is shown that the proposed hardness-model, in combination with the proposed isothermal transformation model, is in agreement with the observed behavior. Amongst others it is successfully predicted that the isothermal transformation precedes the recrystallization of the retained austenite and that the post-heat treatment grain size has a large effect on the behavior through the Hall-Petch effect.

1. Introduction
Laser heat treatment is becoming increasingly adopted in industry for its ability to locally adjust material properties where needed. It offers product designers using austenitic stainless steel the ability to design products with extreme combinations of strength ranging from 200 to 2200 MPa yield stress in a single product. To realize these material property combinations, the material is subjected to a high temperature (over 650 °C) for a short time (0 - 100 seconds) and allowed to cool back to room temperature before subsequent processing. This leads to ‘martensite ($\alpha'$) to austenite ($\gamma$) reversal’ whereby the cold rolling induced martensite is reverted back to finely grained austenite with excellent strength and ductility [1].

Reverse transformation occurs by two main mechanisms i.e. diffusionless ‘shear-type’ (athermal) and diffusional (isothermal). The type of transformation is paramount for the resulting mechanical behavior since it determines the structure and characteristics of the reverted austenite. During diffuse $\alpha'$ to $\gamma$ transformation, equiaxed finely grained austenite is formed with low dislocation density whereas diffusionless transformation produces lath-shaped austenite with a high dislocation density [2].
To be able to design the desired product properties and the processes required to produce them, it is essential to be able to predict the heat treatment induced room-temperature material properties. Rajasekhara has presented a model for the post-heat treatment mechanical properties of AISI 301LN taking into account contributions to the yield stress like precipitation hardening and solid solution strengthening [3]. Furthermore several models were presented to account for diffusional reversion [4–6]. However these models either model the reverse transformation or the resulting mechanical behavior and not the combination of the two. In this work a new model will be presented which predicts both the transformation and the post-heat treatment hardness. The model is designed around the three main hardness-affecting mechanisms being the isothermal transformation of martensite to austenite, static recrystallization of retained austenite and grain boundary strengthening by grain refinement of austenite.

2. Experiments
The laser heat treatments were conducted on commercially available 1 mm thick AISI 301 FS sheet of the company ZAPP, tensile strength 1650 MPa and containing approximately 55% martensite. Heat treatments ranging from 650 °C up to 900 °C for holding times of 0, 10 and 100 s were performed to characterize the reverse transformation. Before and after each heat treatment the ferrite content was measured using a Ferritescope (Fisher MP30) employing the correction-factor as proposed by Petersen [7]. The specimens were laser machined without significant heat affected zone. The tensile area of the specimen (see Figure 1) was irradiated with a fiber coupled 2 kW Diode Laser (DF020 HQ - ROFIN) using a custom diffractive optical element (DOE) producing a rectangular top-hat intensity distribution. This ensures a large uniform temperature zone at the center of the specimens.

A type-k thermocouple was spot welded on the back side of the sample at the center location of the irradiated spot serving as a feedback for the PID-control of the heat treatment. After reaching the desired temperature and holding it for the desired time the sample was water quenched. The typical heating rate and cooling rate was 350 K/s and 4000 K/s respectively, an example is depicted in Figure 2. At this heating rate the thermal gradient over the thickness was measured to be 30 °C.

After heat treatment a LECO micro-indenter type LM 300 AT was used to perform Hardness Vickers measurements on the perpendicular cross section of the heat treated area. In this work the relation of Tabor [8] is employed to convert Hardness Vickers to Yield Stress ($HV = 3\sigma_y/9.8$).

3. Modeling and Results
In this section the proposed model is elaborated starting with the reverse transformation, followed by the relation for the composite yield stress and ending with the model for the austenite yield stress, containing contributions from the static recrystallization of the retained austenite and grain boundary strengthening of the isothermally formed austenite and retained austenite.

3.1. Isothermal Transformation
In 2009 Somani has investigated the relation between alloying content and the possibility of athermal transformation and showed that AISI 301 could revert athermally provided that the heating rate is sufficient [5]. However in the current investigation a less stable AISI 301 is investigated and the chromium and nickel equivalents of this grade, in combination with the heating rate, are such that it is expected that athermal transformation is negligible and therefore only isothermal transformation is considered in the model. Isothermal transformation is governed by nucleation and growth. Leblond et al. have described an excellent model for isothermal transformation [9]. This model will be employed here to describe the reverse
transformation of martensite to austenite. The model of Leblond explicitly accounts for the possibility of a temperature dependent isothermal transformation saturation \( \varphi_{\gamma}^\gamma(T) \). This is something that is also observed for isothermal transformation within industrially interesting time and temperature scales. The rate dependent isothermal transformation is described by:

\[
\frac{d\varphi_{\gamma}^\gamma}{dt} = \frac{\varphi_{\gamma}^\gamma(T) - \varphi_{\gamma}^\gamma}{\tau(T)}
\] (1)

Where the temperature dependence of time constant \( \tau \) is described by an Arrhenius temperature dependence:

\[
\tau(T) = \tau_0 \exp \left( \frac{Q_\tau}{RT} \right)
\] (2)

Where \( Q_\tau \) is the activation energy for isothermal transformation. The temperature dependence of the isothermal transformation saturation is described by an exponential function:

\[
\varphi_{\gamma}^\gamma(T) = 1 - \exp \left( -a_{sat}(T - A_{sat}) \right); T > A_{sat}
\] (3)

3.2. Austenite Martensite Composite Yield Stress

The yield stress of the composite is a mixture of the yield stress of martensite and austenite. Depending on the extent of isothermal transformation the yield stress will be dominated by one of the two and also the relation between composite yield stress and the stress of the individual phases should account for the fact that the martensitic phase is in general much harder than the austenitic phase. Geijsselaers has derived an approximation considering a ‘hard’ martensitic phase inside a ‘ductile’ austenitic matrix [10].

\[
\sigma = \varphi^\gamma \sigma_{\gamma}^\gamma + f(\varphi^{\alpha'}) \sigma_{\alpha'}^{\alpha'}
\] (4)

Where \( f(\varphi^{\alpha'}) \) is determined by the current fraction of martensite and the ratio of austenite to martensite yield stress \( (C = 1.383\sigma_{\gamma}^\gamma/\sigma_{\alpha'}^{\alpha'}) \):

\[
f(\varphi^{\alpha'}) = \varphi^{\alpha'} \left( C + 2(1-C)\varphi^{\alpha'} - (1-C)\varphi^{\alpha'}^2 \right)
\] (5)
3.3. Austenite Yield Stress
The yield stress of the austenite phase is considered to be the weighted average of the yield stress in the retained austenite fraction $\phi^\gamma R$ and isothermally formed fraction $\phi^\gamma I$.

$$\sigma_y^\gamma = \frac{\phi^\gamma R}{\phi^\gamma} \sigma_y^\gamma R + \frac{\phi^\gamma I}{\phi^\gamma} \sigma_y^\gamma I$$  \hspace{1cm} (6)

As mentioned in the introduction the retained austenite is prone to SRX, also the SRX induces a grain refinement causing an increase in yield strength by grain boundary strengthening effects. This can be modeled as proposed by amongst others Jonas [11] and a Hall-Petch term respectively.

$$\sigma_y^\gamma R = \sigma_y^\gamma R_0 - f_{srx}(\sigma_y^\gamma R_0 - \sigma_y^\gamma_0) + \frac{K}{\sqrt{d_R}}$$  \hspace{1cm} (7)

Where $\sigma_y^\gamma R_0$ is the yield stress of the retained austenite prior to heat treatment and $\sigma_y^\gamma_0$ is the yield stress of the recrystallized austenitic material, i.e. with all work hardening removed. $K$ is the Hall-Petch coefficient. The magnitude of the Hall-Petch contribution to the yield stress depends on the evolution of the grain size of the retained austenite, this is modeled by linking the grain size evolution to the recrystallization.

$$d_R = d_{R_0} - f_{srx}(d_{R_0} - d_{R_{ss}})$$  \hspace{1cm} (8)

To complete the model for the prediction of the weighted average austenite yield strength a relation is needed for the yield strength of the isothermally formed austenite. Upon isothermal transformation new austenite grains nucleate and grow, i.e. the highly dislocated martensite is replaced by dislocation-free new austenite grains. The size of the resultant grains is very small (in the sub-micron range) and thus a significant Hall-Petch strengthening effect must be accounted for:

$$\sigma_y^\gamma I = \sigma_y^\gamma_0 + \frac{K}{\sqrt{d_I}}$$  \hspace{1cm} (9)

The last ingredient for the determination of the yield strength of the retained austenite is an equation for the time and temperature dependent SRX [12].

$$f_{srx} = 1 - \exp\left(-.693\left(\frac{t}{t_{0.5}}\right)^n\right)$$  \hspace{1cm} (10)

Where $t_{0.5}$ is the temperature-dependent time required for 50% recrystallization of the material, for static recrystallization it can be described by:

$$t_{0.5} = k_0 \exp\left(\frac{Q_{srx}}{RT}\right)$$  \hspace{1cm} (11)

Here $k_0$ is related to the prior deformation history and the grain size when the recrystallization process starts and $Q_{srx}$ is the activation energy for static recrystallization.

3.4. Parameter Identification and Results
The model features in total 14 parameters of which 4 were fitted to the transformation data in Figure 3 ($\tau_0$, $Q_\tau$, $a_{sat}$ and $A_{sat}$), 4 were fitted to the Hardness Vickers data in Figure 4 ($K$, $k_0$, $Q_{srx}$ and $n$) and 6 were selected based on literature. The fitting was performed using a least squares optimization, the parameters are presented in Table 1. The experimental and modeling results and a SEM-picture displaying isothermally reverted grains is shown in Figures 3-6.
\[ \tau_0 = 2.24 \cdot 10^{-11}\text{ s} \quad \sigma^r_{y0} = 350\text{ MPa} \quad d_I = 0.2\text{ \textmu m} \]
\[ Q_\tau = 209\text{ kJ/mol} \quad d^r_{y0} = 1700\text{ MPa} \quad k_0 = 6.86 \cdot 10^{-10}\text{ s} \]
\[ a_{sat} = 0.01\text{ K}^{-1} \quad \sigma^r_{y0} = 1183\text{ MPa} \quad Q_{srx} = 214.4\text{ kJ/mol} \]
\[ A_{sat} = 811\text{ K} \quad d_{R0} = 15\text{ \microm m} \quad n = 1.29 \]
\[ K = 357\text{ MPa \mu m}^{1/2} \quad d_{Rss} = 3\text{ \microm m} \]

**Table 1: Model parameter values.**

**4. Discussion and Conclusion**

In this work laser heating experiments were presented to characterize the effect of fast heat treatment on the reverse transformation from martensite to austenite and to link this transformation to the resulting mechanical behavior. A model was presented which takes several important mechanisms into account like isothermal transformation, recrystallization and grain boundary...
strengthening. First, the transformation model was fitted to the experimental transformation data. Next, these results were transferred to the model for the prediction of hardness which was fitted to the hardness data from the experiments. Figure 3 shows a good agreement between the experiments and the prediction of isothermal transformation. Also the necessity of a temperature dependent transformation saturation is obvious, i.e. at 800 °C there is practically no reverse transformation between 10 and 100 s. In Figure 4 the prediction of hardness is displayed versus the measurements. Clearly there is a good qualitative agreement between model and experiments, this is a good indication that the model captures the main mechanisms dominating the post-heat treatment mechanical properties. From the HV model-results it seems that the biggest discrepancies between predicted and experimental HV stem from a mismatch between the predicted isothermal and experimental transformation amount. Indeed at e.g. 750 degrees and 10 seconds holding time the isothermal transformation amount is significantly overestimated and subsequently (and consistently) the HV-value is underestimated.

A second model validation is depicted in Figure 5. Here the hardness is plotted against the evolution of the austenite fraction. There is a clear trend which shows a relation between the amount of transformation and the decrease in Hardness Vickers. Only after transformation is (almost) complete, does recrystallization start to play a (significant) role, in agreement with the observation of Kisko et al. that transformation precedes recrystallization during phase reversal of austenitic stainless steel [1].

In the current model the Hall-Petch contribution to the overall yield stress is quite significant, especially for the isothermally formed austenite (\(K_d/\sqrt{d_I} \approx 750 \text{ MPa}\)). This is in agreement with the experimental results of Di Schino [13] and Huang [14] who found large Hall-Petch effects for the finely grained reverted austenite in AISI 301 and AISI 301LN respectively. The magnitude of the Hall-Petch effect is determined by the size of the isothermally reverted grains which, for the isothermally formed austenite, has been implemented as a ‘constant’ of 0.2 µm. The SEM picture (Quanta FEG 650) shown in Figure 6 on the completely reverted microstructure of a sample heated to 900 °C for 0 s reveals a large number of very small ‘reverted’ grains alongside larger ‘retained grains’. The size of the reverted grains is indeed in submicron to micron range.

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