Flow Curve Modelling of an Mg-PSZ Reinforced TRIP-matrix-composite

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A prediction model for cold flow curves was introduced for a new class of composite materials known as TRIP-matrix-composites consisting of three phases, austenite, strain-induced martensite and ZrO₂. The content of the ceramic phase was varied between 0 and 30%, whereas the particle size of the ceramic was selected to be 10 to 30 μm. For the manufacturing of the composite material the powder metallurgical route including hot press procedure was chosen and very dense material could be produced.

Included in the model is the hydrostatic stress \( \sigma_m \) close to the circumferential surface of the compression test sample. The hydrostatic stress was varied using different material compositions and true strain values. To calculate the cold flow curves the ISO-E-method was applied. The calculated results show a consistent congruency with the experimental data.

KEY WORDS: Metal-matrix-composite; TRIP-steel; ZrO₂; modelling; flow curve; hydrostatic stress.

1. Introduction

The commercial production of complex construction parts or safety-related components is often only possible by the use of new, high strength materials. The main trend is the application of so-called multiphase-materials (in short MP-materials), including MP-steels, which show a promising step to lightweight construction. Their structure is characterized by a combination of different hard and soft phase components. Examples for MP-steels are dual phase steels, so-called residual austenite steels, complex phase steels and martensitic phase steels. One group of the recent MP-steels are TRIP-matrix-composites based on newly developed austenitic cast TRIP-steel material in combination with MgO-partially stabilized ZrO₂ ceramic. This new material had been developed and is under investigation within the collaborative research centre CRC 799.

Both components have the potential of strain-induced martensitic phase transformation. Thus they are able to compose local excessive stress. The basic concept of this metal-matrix-composite is the possibility to induce mechanical phase transformation by custom-made interfaces between structural constituents. Beside the infiltration of ceramic macrostructures by steel melts the powder metallurgy is another way of manufacturing these TRIP-matrix-composites in which all scopes of design are permitted. The authors had already published results for consolidation of these composites by means of conventional and conductive sintering as well as powder forging and hot pressing. It is obvious that for a given material composition the properties are not only influenced by residual porosity but also by destabilization of ZrO₂ during the manufacturing process, with constant thermic and mechanical load (Fig. 1). To obtain a robust process layout by means of FEM it is necessary to reproduce the significant process properties also by model calculations. On the other hand, the development of experimental methods for measuring the forming and failing behaviour is required. On the other hand, a mathematical model has to be designed, using the parameters phase transformation, flow properties and fracture.

To forecast the flow properties of steels with TRIP-effect a model is required using plastic deformation considering martensite formation. Different models are known from the literature. One essential part of these models is a module calculating the content of residual austenite or martensite as a function of true strain. The modelling of the flow curves itself both of structural phases and multi component materials are based on the general mixture rule by Tamura.

Fig. 1. Properties of TRIP-matrix composites containing 5% ZrO₂: hot pressed (1 250°C/1 h/30 MPa), conventional sintered (1 420°C/2 h/vacuum) and conductive sintered (50 K/6 min/40–15 MPa).
\[
\sigma_{\text{mix}} = f_\alpha \sigma_\alpha (\varepsilon_\alpha) + f_\beta \sigma_\beta (\varepsilon_\beta), \quad \ldots (1)
\]
\[
\varepsilon_{\text{mix}} = f_\alpha \varepsilon_\alpha + f_\beta \varepsilon_\beta. \quad \ldots (2)
\]

The mixture rule indicates that the stresses, as well as the strains, can be divided in sums of stresses, respectively strains, of the single components multiplied by the volume fraction of these components.

The so called ISO-E-method is the further development of the models mentioned above.\(^{10}\) The ISO-E-method claims that for a disordered microstructure, the energy density increment can be equated for each constituent, independent of the material state. The ISO-E-method is expressed as follows:

\[
\int_0^{\varepsilon_f} \sigma_{\text{mix}} (\varepsilon) d\varepsilon = \int_0^{\varepsilon_f} \sigma_\alpha (\varepsilon) d\varepsilon + \int_0^{\varepsilon_f} \sigma_\beta (\varepsilon) d\varepsilon + \ldots \int_0^{\varepsilon_f} \sigma_n (\varepsilon) d\varepsilon = \ldots . (3)
\]

It is state of the art that for prediction of the flow properties of TRIP-matrix composites only the model for the matrix material (CrMnNi TRIP-steel) is used.\(^{12,13}\) For this reason the aim of this article is the application and modification of existing models for flow curve prediction of MP-materials for the new composites. The model quality, as well as the accuracy of the prediction, has to be significantly increased. For the modelling it is required that very densely sintered TRIP-matrix-composites are used with completely destabilized ceramic fraction during the manufacturing process.

### 2. Experimental Details

The investigated material is based on two components, on the one hand, there is a manganese-containing austenitic gas-atomized steel powder \((d_{10} = 8 \mu m, d_{50} = 20 \mu m \text{ and } d_{90} = 127 \mu m)\). On the other hand, there is a powder of MgO-partially stabilized ZrO\(_2\) ceramic \((d_{10} = 11 \mu m, d_{50} = 18 \mu m \text{ and } d_{90} = 30 \mu m)\). The chemical composition of the metastable high alloyed TRIP-steel (indicated as 16-7-6 with the nominal concentration of Cr, Mn and Ni), the stable austenitic steel AISI 316L\(^{12,13}\) and the ZrO\(_2\) ceramic are shown in Table 1. The stable austenitic steel AISI 316L is needed for the modelling of own experimental results.

By varying the ceramic particle content five different powder mixtures were obtained. Therefore, the contents of ceramic particles in the steel matrix were given by 0%, 5%, 10%, 20% and 30%. These mixtures were cold pre-compressed into disc form with a diameter of 155 mm and a height of 36 mm using uniaxial pressure with 90 MPa. Finally the samples were hot pressed and sintered at 1 250°C for 30 min under protective gas Varigon, consisting of 5% H\(_2\) and 95% Ar. The heating and cooling rates were 10 K/min, respectively. Afterwars a set of specimens was selected for the metallographic examination and visualization of the particle distribution of the ZrO\(_2\) in a TRIP-steel matrix (Figs. 2(a)–2(d)). TRIP-steel regions are shown in light grey and the ZrO\(_2\) in dark grey. It is obvious that the particle distribution in the TRIP-steel matrix is nearly homogenous and there were no agglomerates found.

The porosity of each sample was measured using the Archimedes method. It was observed that the samples are almost full dense with the values of the total porosity of at most 0.1%. For this reason it was stated that the subsequent tests were conducted with compact material states.

Cold upsetting specimens of 9 mm in initial diameter and 16.2 mm in initial height, corresponding to an aspect ratio of 1.8, were prepared by machining from hot pressed discs. The measurements such as height \((h_0)\) and diameter \((d_0)\) were carried out before the deformation. The cold upsetting specimens were deformed at room temperature between flat dies using incremental strain steps of 0.05, increased until cracks were visible to the naked eye on the free surface of the preform. The strain rate amounts to 0.1 s\(^{-1}\). The die surfaces were mirror polished. During the upsetting tests the die set was well lubricated by oil. After each interval of loading dimensional changes in the specimen such as height \((h_0)\), top contact diameter \((d_c)\), bottom contact diameter \((d_b)\) and bulged diameter \((d_b)\) were measured using a light measuring microscope. The schematic view of the preform before and after deformation is shown in Fig. 3.

In order to evaluate the kinetics of the \(\alpha’\)-martensite evolution for the TRIP-steel 16-7-6, the ferromagnetic phase fraction of the samples with different contents of ceramic

![Fig. 2. Distribution of second-phase particle of ZrO\(_2\) in TRIP-steel-5% ZrO\(_2\) (a), TRIP-steel-10% ZrO\(_2\) (b), TRIP-steel-20% ZrO\(_2\) (c) and TRIP-steel-30% ZrO\(_2\) (d).](image1)

![Fig. 3. Upset-forging test preform, before and after deformation.](image2)
particles were measured using magnetic balance testing and a previous calibration. The measuring principle is based on a magnetic induction method. In the used material ferromagnetic phases such as the δ-ferrite and α'-martensite can be found.\(^\text{12}\) As known from literature, the presence of the martensitic hcp phase of iron\(^\text{14}\) is not expected at Mn contents below 11 wt.-%. Furthermore, the low temperatures and low strain rates lead to the formation of ε-martensite within deformation bands from the austenite and transform into α'-martensite during deformation (TRIP).\(^\text{15}\) At the beginning of each experiment, i.e. in α'-martensite-free state, the amount of δ-ferrite was determined to extract these contents from the measurement values of the experiment and thus to obtain the exact amount of α'-martensite.

3. Theoretical Analysis

The mathematical expressions used and proposed for the determination of various upsetting parameters under plane stress state conditions (\(ε = 0\)) in the vicinity of the circumferential surface of the upsetting specimen are discussed below.

According to Abdel-Rahman and El-Sheik,\(^\text{16}\) the expression for the axial strain (\(ε_z\)) can be written as follows:

\[
e_\tau = \ln \frac{h_\tau}{h_0}, \quad \text{............... (4)}
\]

where \(h_0\) is the initial height and \(h_\tau\) is the deformed height of the preform.

According to Narayanasamy and Pandey\(^\text{17}\) the expression for the hoop strain (\(ε_θ\)) is given by:

\[
e_\theta = \ln \frac{2d_c^2 + d_z^2}{3d_0^2}, \quad \text{............... (5)}
\]

where \(d_0\) is the bulge diameter, \(d_\theta\) is the initial diameter and \(d_z\) is the average contact diameter of the preform. The average diameter of the preform is expressed as:

\[
d_z = \frac{d_\tau + d_\infty}{2}, \quad \text{............... (6)}
\]

In the above expression (6) is \(d_\tau\) the contact diameter of the preform at the top and \(d_\infty\) the contact diameter of the preform at the bottom.

The true axial stress \(σ_z\) is defined as:

\[
σ_z = \frac{F}{A_\tau}, \quad \text{............... (7)}
\]

where \(F\) is the load and \(A_\tau = \frac{\pi d_\tau^2}{4}\) is the contact area.

The true stress in the hoop direction \(σ_θ\) is according to Narayanasamy et al.\(^\text{\text{18}}\):

\[
σ_\theta = \frac{1 + 2ν}{2 + ν} σ_z, \quad \text{............... (8)}
\]

where \(ν = \frac{ε_r}{2ε_z}\) is Poisson’s ratio.

The true hydrostatic stress (\(σ_m\)) is than calculated by:

\[
σ_m = \frac{σ_z + 2σ_θ}{3}, \quad \text{............... (9)}
\]

Usually \(σ_z\) is negative because it is compressive in nature and \(σ_\theta\) is positive.

4. Results and Discussions

The stress-strain diagram of the single components of the composite–TRIP-steel 16-7-6, its stable austenitic variation AISI 316L, 
ZrO\(_2\) and the strain-induced martensite–are illustrated in Fig. 4. The datasets originate from different sources but being determined under similar experimental conditions: TRIP-steel data are own investigations, ceramic data comes from the IKTS in Dresden, AISI 316L is part of\(^\text{12}\) and martensite data are given in.\(^\text{19}\) The expected results for the strain-induced martensite exhibits very good congruence with the results of\(^\text{12}\).

Pyshmintsev et al.\(^\text{20}\) examined the influence of the hydrostatic compressive strength \(σ_{\text{HS}}\) on the stability of the residual austenite, being loaded to tensile specimen during tensile tests. Therefore, a special technology was applied using a box with a hydrostatic compression generator. The external compression strength during tensile tests was varied between 0 and 800 MPa. The authors described the following model equation, combining the fracture content of still not transformed residual austenite \(f_r\), the true strain \(ε_z\) as well as the hydrostatic compressive strength \(σ_{\text{HS}}\):

\[
f_r = f_r^{(0)} \exp\left(-\left[p - z \cdot σ_{\text{HS}}\right] \cdot ε_z\right), \quad \text{............... (10)}
\]

where \(f_r^{(0)}\) means the content of residual austenite and \(p\) and \(z\) are empirical coefficients.

In order to adapt the literature model to the own used experimental study of “pure” compression test the external compression strength \(σ_{\text{HS}}\) has to be replaced by the resulting hydrostatic stress \(σ_m\) located close to the circumferential surface of the compression test sample. It is changing with the proceeding strain-induced \(γ→α'\)-transformation due to the expanded volume of the martensite compared to austenite and also due to the different ZrO\(_2\) contents.

The hydrostatic stress \(σ_m\) calculated according to Eqs. (4) to (9) is represented in Fig. 5 for the investigated material compositions. The hydrostatic stress is illustrated, which is in fact, negative. For a ZrO\(_2\)-content of 20% or more a significant change in the curve progression is obvious. For providing best congruence between calculated and experimental data a mathematical relationship was looked for, beginning with 0\%, 5\% and 10\% ceramic content. The same procedure was done for 20\% and 30\% separately. It becomes apparent that a power law results in the best fit of calculated and experimental dataset.

**Fig. 4.** Compressive stress-strain diagrams of ZrO\(_2\), α’-martensite, TRIP-steel 16-7-6 and a stable steel AISI 316L at 20°C.
The program TableCurve was used to compute the optimal values of \( p \) and \( z \) to minimize the deviation between model and experimental values of the residual austenite. Therefore, the measured data for the hydrostatic stress \( \sigma_{HD} \) were inserted into Eq. (10) replacing \( \sigma_{HD} \). For this iterative process the starting points for \( p \) and \( z \) were taken from the literature.20)

After the parameters \( p \) and \( z \) were identified the residual austenite values after the first four incremental strain steps and after crack were calculated for all investigated specimen. The comparison of the measured and the calculated values by the modified model of Pyshmintsev et al.20) are illustrated in Fig. 6. It is evident that the difference between the modified and the experimental values increases with proceeding strain-induced \( \gamma \rightarrow \alpha' \)-transformation. For the quantification of the model quality of the modified equation the root mean square (RMS) between calculated and experimental values was used.

\[
RMS = \sqrt{\frac{\sum (x_{i,\text{meas}} - x_{i,\text{calc}})^2}{n}} .......... (11)
\]

where value \( x_{i,\text{meas}} \) is the measured and \( x_{i,\text{calc}} \) is the belonging calculated value, \( n \) is the number of pairs of variates. The specific prediction accuracy of the correlation in percent was calculated by dividing \( RMS \) by the arithmetic average \( \bar{x} \) of \( x_{i,\text{meas}} \).

\[
\bar{x} = \frac{1}{n} \sum x_{i,\text{meas}} .......... (12)
\]

and multiplying it by 100. The value of RMS come to 0.078 being calculated from the modified Eq. (10) using the calculated residual austenite contents, when \( p = -0.167 \) and \( z = 0.0069 \). For an arithmetic average of \( \bar{x} = 0.78 \) the specific deviation is 10%. This means that for the residual austenite content of e.g. 10% the absolute prediction accuracy is ±1%.

For the ceramic contents 0%, 5% and 10% a generalized power function \( \sigma_{m} = f(\varepsilon) \) was used and the specific deviation is only about 3% higher (RMS = 0.106 and \( \bar{x} = 0.78 \)). The modified model exhibits a good accuracy for the investigated composite.

According to the general mixture rule by Tamura et al.10) (Eqs. (1) and (2)) the flow stress \( \sigma_{ZrO_2} \) of the composite is defined as

\[
\sigma_{z} (\varepsilon_{z}) = f_{z} (\varepsilon_{z}) \cdot \sigma_{\gamma} (\varepsilon_{\gamma}) + f_{\alpha'} (\varepsilon_{\alpha'}) \cdot \sigma_{\alpha'} (\varepsilon_{\alpha'}) + f_{ZrO_2} \cdot \sigma_{ZrO_2} (\varepsilon_{ZrO_2}) , .... (13)
\]

If all structural phases are considered, then

\[
f_{z} (\varepsilon_{z}) + f_{\alpha'} (\varepsilon_{\alpha'}) + f_{ZrO_2} = 1 .......... (14)
\]

hold. Assuming that the energy density has the same value for the single structural constituent as well as for the whole sample at every moment of the deformation (the so called ISO-E-hypothesis), the flow curves of the composites were calculated (Fig. 7). The modified model for the kinetics of the strain-induced transformation of the residual austenite being described above was implemented. The calculation of one point of the flow curve of the composite executes in the following way: First, a strain in ZrO2 was specified and the corresponding energy density of the stress-strain curve (Fig. 4) was determined. Second, for the phases \( \alpha' \) and \( \gamma \) those strain was iteratively searched which satisfies the condition in Eq. (3). Third, using Eq. (2), the strain of the composite was calculated. Finally, for the calculated composite strain the corresponding composite stress according to the Eq. (3) was searched iteratively. The results exhibit very good accuracy between the calculated and measured datasets. The specific prediction accuracy of the correlation according to Eqs. (11) and (12) is 2%, 2% and 1% for the chosen ZrO2-content of 0%, 10% and 30%, respectively. Our effort to find a fast and easy method for prediction of flow curves of TRIP-

Fig. 5. The variation of hydrostatic stress with respect to the true axial strain for different ZrO2 percentage.

Fig. 6. Comparison of measured values and calculated values based on the modified model by Pyshmintsev et al.20) (Eq. (10)) for the residual austenite content given by constant \( p = -0.167 \) and \( z = 0.0069 \).

Fig. 7. Predicted flow curves of the TRIP-matrix composite with 0%, 10% and 30% ZrO2 using calculated values for the strain-induced transformation of residual austenite to martensite.
matrix composite can be considered successful. This is essential for a subsequent simulation.

5. Conclusion

The deformation behaviour of a TRIP-Matrix-Composite with the MgO-partially stabilized ZrO$_2$ content between 0% and 30% was investigated in order to model the flow stress. Therefore, the material behaviours of the single components, austenite, martensite and ceramic ZrO$_2$ were used. At various strains the hydrostatic stress in the vicinity of the circumferential surface of the upsetting specimen was measured. Finally, the compressive flow stress of the TRIP-Matrix-Composite was calculated using the ISO-E-Method. In this calculation, the measured flow stress data of the martensite, the stable austenite AISI 316L and of the ceramic ZrO$_2$ were used. The following conclusions can be drawn from the above results and discussions:

- The model of Pyshmintsev et al.\textsuperscript{20} for calculation kinetics of strain-induced transformation of residual austenite in low alloyed TRIP-steels, was modified for TRIP-matrix composites. The modification is based on measurements of hydrostatic stress $\sigma_m$ close to the circumferential surface of the compression test sample. The specific prediction accuracy of the calculated values is according to the residual austenite content $\pm 2\%$.
- It is obvious that a significant change of hydrostatic stress $\sigma_m$ close to the circumferential surface starts, when ZrO$_2$-content is $20\%$ or higher. For that reason a power function $\sigma_m = f(\epsilon)$ was used for calculating the flow stress of the ceramic contents $0\%$, $5\%$ and $10\%$. The specific deviation is only about $3\%$ higher and hence acceptable.
- Knowing the flow curves of the three single structural phases and using the ISO-E-method and the general mixture rule by Tamura\textsuperscript{10} it was possible to calculate the flow curves for the composite. The calculated and measured values are nearly congruent and exhibit a specific accuracy of not more than $\pm 4\%$.

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Acronym

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
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<tbody>
<tr>
<td>CRC</td>
<td>Collaborative research centre</td>
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<tr>
<td>FEM</td>
<td>Finite element method</td>
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<tr>
<td>IKTS</td>
<td>The Fraunhofer Institute for Ceramic Technologies and Systems</td>
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<tr>
<td>ISO-E-method</td>
<td>Iso-energy method</td>
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<tr>
<td>Mg-PSZ</td>
<td>Zirconium dioxide partially stabilized with MgO</td>
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<td>MMC</td>
<td>Metal-matrix-composite</td>
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<tr>
<td>MP</td>
<td>Multiphase</td>
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<tr>
<td>RMS</td>
<td>Root mean square</td>
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<tr>
<td>TRIP</td>
<td>Transformation induced plasticity</td>
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</table>

Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$f$</td>
<td>Volume fraction of a phase</td>
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<tr>
<td>$\gamma$</td>
<td>fcc phase of iron</td>
</tr>
<tr>
<td>$\delta$-ferrite</td>
<td>Bcc phase of iron</td>
</tr>
<tr>
<td>$\alpha$-martensite</td>
<td>Martensitic bcc phase of iron</td>
</tr>
<tr>
<td>$\epsilon$-martensite</td>
<td>Martensitic hcp phase of iron</td>
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<tr>
<td>$h_0$</td>
<td>Initial height of the cylindrical preform</td>
</tr>
<tr>
<td>$h_l$</td>
<td>Height of the barrelled cylinder after deformation</td>
</tr>
<tr>
<td>$d_0$</td>
<td>Initial diameter of the preform</td>
</tr>
<tr>
<td>$d_b$</td>
<td>Bulged diameter of the preform after deformation</td>
</tr>
<tr>
<td>$d_{bc}$</td>
<td>Top contact diameter of the preform after deformation</td>
</tr>
<tr>
<td>$d_{dc}$</td>
<td>Bottom contact diameter of the preform after deformation</td>
</tr>
<tr>
<td>$\langle \sigma \rangle$</td>
<td>Average contact diameter of the preform</td>
</tr>
<tr>
<td>$\nu$</td>
<td>Poisson’s ratio</td>
</tr>
<tr>
<td>$F$</td>
<td>Force applied on the cylindrical preform for deformation</td>
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<tr>
<td>$\sigma_x$</td>
<td>True stress in the axial direction</td>
</tr>
<tr>
<td>$\sigma_y$</td>
<td>True stress in the hoop direction</td>
</tr>
<tr>
<td>$\sigma_z$</td>
<td>True stress in the radial direction</td>
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<tr>
<td>$\sigma_m$</td>
<td>True hydrostatic stress</td>
</tr>
<tr>
<td>$\sigma_{\text{ISO}}$</td>
<td>Exterior hydrostatic compression strength during tensile test</td>
</tr>
<tr>
<td>$\epsilon_x$</td>
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<tr>
<td>$\epsilon_y$</td>
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REFERENCES