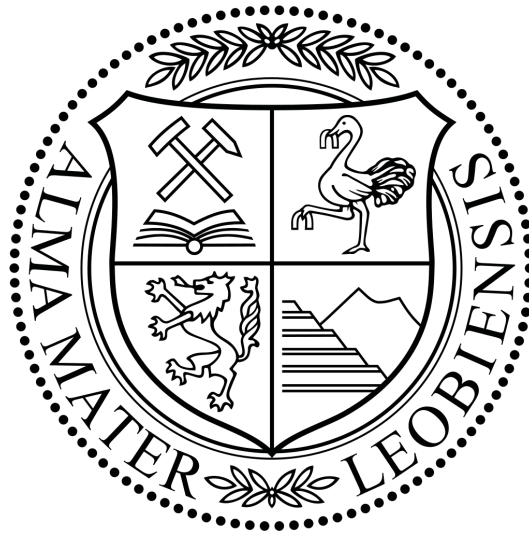




RESEARCH PAPER

Melanie Tomasch, Dipl.- Ing.

**Fundamentals and prevention of zinc induced embrittlement
in steel sheets during the direct press hardening process**

PROF. DR. THOMAS ANTRETTTER

MONTANUNIVERSITÄT LEOBEN

PROF. DR. RAJIV SHIVPURI

THE OHIO STATE UNIVERSITY

LEOBEN SEPTEMBER 24, 2018

Statutory declaration

I declare in lieu of oath, that I wrote this research paper and performed the associated research myself, using only literature cited in this volume.

Melanie Tomasch

Leoben September 24, 2018

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Abstract

Press hardening of low alloy steel sheets is an efficient way of manufacturing high strength automotive components relevant for passenger safety in case of a crash. The press hardening process combines the advantages of good formability at elevated temperatures as well as high strength and good form accuracy after quenching in the closed die. To guarantee corrosion protection of the components during the in service time a zinc coating is applied on the entire coil surface by hot dip galvanizing. On the one hand this ensures a continuous coating thickness but on the other hand the coating has to withstand the heating, forming and quenching cycle. Cracks with a length of up to 200 microns occurred in the past during the manufacturing process. The zinc coating which is still liquid after removing the specimen from the oven is assumed to cause the harmful cracking of the base material. The phenomenon leading to this kind of cracks during the forming process is called liquid metal embrittlement (LME).

A numerical model of the direct press hardening process considering all thermal and mechanical material properties and interactions with the environment and the forming dies was developed. This should subsequently help to identify and understand the mechanisms and influencing factors leading to LME.

Since cooling during forming and subsequent quenching has a significant influence on the mechanical behaviour of a steel sheet, a strongly coupled thermo-mechanical model has been developed for the simulation software ABAQUS. Accurate simulation results require the knowledge of temperature dependent thermal and mechanical material properties as well as occurring interactions between the sheet and the die. They were determined in a series of experiments and provide the input for the numerical model. During the quenching step in the closed die, the high strength of the component is achieved by a martensite phase transformation. This transformation is strongly influenced by the deformation in the heavily bent areas as well as the temperature evolution of the sheet. Accurate results for the shape of the final components and for the properties within the operating service time can only be obtained if the numerical model accounts for the microstructure formation including the TRIP (Transformation Induced Plasticity) effect. This is accomplished by means of a user subroutine. Consequently, the model allows to analyse the forming and cooling process in the closed die in detail throughout the entire process cycle. Concluding, the final geometry, the

wall thickness distribution of the component and the press forces during forming are used as parameters to compare the simulation results with forming experiments and to validate the chosen material model. At the end the influencing factors, which favour the phenomenon of LME should be clarified. A numerical simulation model should be able to predict the occurrence of LME during a given direct press hardening process. If necessary the process parameters can be modified in advance and the number of required experiments can be reduced by means of the simulation model.

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Chapter 1

Introduction

Nowadays the passenger safety and fuel efficiency requirements in the automotive industry are steadily increasing due to customer demands as well as legal specifications. One possible way to decrease the fuel consumption and consequently the environmental impact caused by emissions would be the weight reduction of the body in white of the car. However to assure the passenger safety during a crash it is not possible to significantly reduce the thickness of the commonly used steel components of the body in white. A compromise between weight reduction and passenger safety has to be found. This can be achieved by adapting the components geometry or material in a manner that each component and the entire assembly can fulfill exactly its special purpose in case of a crash. On the one hand crash relevant parts must show high strength, on the other hand also good energy absorption behaviour to mitigate the consequences of an accident for the passengers. Due to the vehicle safety and crash requirements as well as the need to enable a cost and time efficient production process in the automotive industry, the use of ultrahigh-strength steels for structural and safety components is rapidly increasing. Ultrahigh-strength steels, however, have a very limited formability at room temperature and show high spring back behavior. Therefore complex part geometries can be manufactured only at elevated temperatures. One concept for the manufacturing of high strength automotive components is the press hardening of low alloy steel sheets. The press hardening process combines the advantage of the good formability at elevated temperatures and the high strength and good form accuracy after the quenching in the closed die.

Another important requirement is the corrosion resistance of the car body. Uncoated steels would show scaling during the production process and will be damaged by corrosion during the life time cycle. To prevent the steel parts from corrosion an AlSi coating can be used. This type of coating unfortunately provides only a barrier corrosion protection of the components. In case of a damaged coating the corrosion protection at that position is not available anymore. An-

other possibility is a zinc based coating. In contrast to the AlSi coating the Zn coating provides a cathodic corrosion protection. This is a great advantage because even in the case of a damaged coating the steel substrate is protected by the surrounding zinc. To ensure a continuous coating thickness the material is already coated before the forming and heat treatment by hot dip galvanizing. Due to the low melting point of pure zinc, the coating becomes liquid during the heat treatment of the part. Depending on the forming temperature still liquid zinc phases might be present on the sheet surface during the forming step. These liquid zinc phases are assumed to be responsible for an embrittlement of the base material leading to cracks during the forming process at elevated temperatures. The phenomenon is called liquid metal embrittlement and will be abbreviated with LME in the following. The purpose of this work was to conduct an experimental as well as numerical investigation of the press hardening process to analyze and understand the conditions leading to LME.

Chapter 2

State of the Art

2.1 Press Hardening

Press hardening enables cost and time efficient manufacturing of form accurate, light weight and high strength components for the body in white of a car relevant in case of a crash. During press hardening low alloy steel sheets are hot formed and quenched in the same die. Due to the elevated temperature during forming, the required press force is reduced and the formability of the material is enhanced. Furthermore the martensite phase transformation occurring during quenching reduces spring back and increases strength which in turn allows to reduce the sheet thickness of the component. Figure 2.1 shows the tensile strength and elongation in the as received and hardened condition [1].

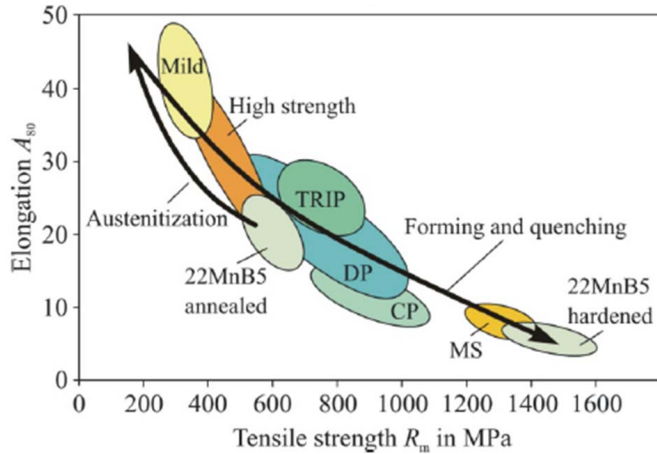


Figure 2.1: Change in tensile strength and total elongation from the as received to the hardened condition. [1]

At the beginning of the direct press hardening process, the sheet metal is heated in the oven from room temperature with ferrite-pearlite microstructure up to approximately 900 °C, where full austenitization occurs. During the subsequent transfer from the oven to the press there is a heat flux due to convection and radiation to the environment. The still austenitic sheet is placed into the water-cooled die and the final form of the component has to be reached at temperatures higher than the martensite start temperature in one single stroke of the press. The schematic outline of the press hardening process is shown in figure 2.2.

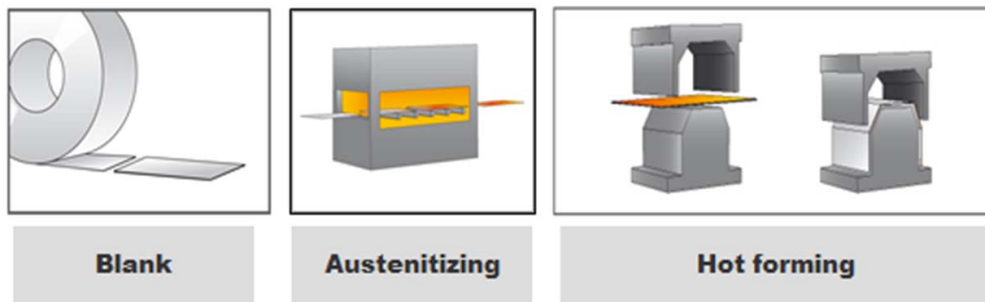


Figure 2.2: Schematic representation of the press hardening process [2].

The formability is highly increased and the press forces, tool wear and spring back are significantly reduced at elevated temperatures. When the specimen has reached its final shape, it is cooled down rapidly in the die with rates higher than approximately 27 °C/s for a 22MnB5 steel according to [1], to enforce a fully martensitic microstructure. The cooling rates are crucial to the final microstructure and thus the final mechanical properties of the part in service. Figure 2.3 shows the temperature evolution and forming step depending on the continuous cooling diagram. The phase transformation from austenite to full martensite leads to a significant increase in tensile strength up to 1500 MPa according to [3, 4]. Press hardening enables cost and time efficient manufacturing of form accurate, light weight and high strength components for the body in white of a car relevant in case of a crash. By means of direct hot forming complex, crash resistant parts such as bumpers, pillars and side impact beams with ultrahigh strength, minimum spring back, and reduced sheet thickness can be produced [3, 4].

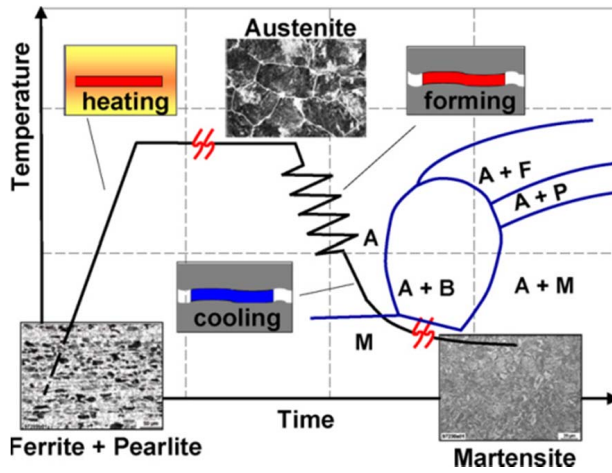


Figure 2.3: Temperature evolution and forming step depicted in a schematic continuous cooling diagram [5].

2.2 Phase Transformation

Heat treatment of materials is used in a wide range of applications since it allows to influence the material's properties like hardness, brittleness, strength and wear resistance to suit particular requirements.

In the case of hot forming the mechanical properties of the steel sheet are changed according to the component's requirements by austenization and subsequent martensitic phase transformation. During the press hardening process the occurring heat flux to the tool and to the ambient air is leading to a temperature decrease in the specimen. Due to this change in temperature the initial austenitic phase decomposes into the product phase martensite. The continuous cooling diagram for a boron manganese press hardenable steel is depicted in figure 2.4. At high temperatures the microstructure is characterized by an austenitic cubic face centered lattice. During cooling a phase transformation occurs due to the minimisation of the free energy of the atoms and a new lattice structure is formed depending on the prevailing temperature and stress-strain conditions as well as the chemical composition of the alloy and the cooling rate. As can be seen the cooling rate must be high enough to ensure martensitic phase transformation and to avoid a transformation to ferrite, pearlite and bainite.

Additionally it is known [7] that the previous forming history has a strong influence on the formed phase which can be seen in a shift of the phase regions in the CCT diagram depicted in figure 2.5.

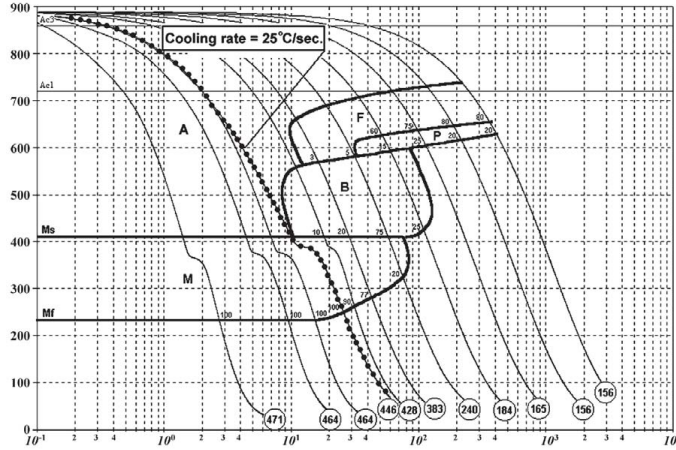


Figure 2.4: Continuous cooling diagram of a 22MnB5 press hardenable steel. [6]

The phase transformation is in general accompanied by a rearrangement of the metal structure, a change in volume, and a heat release. That leads to a new stress strain state leading to distortion and residual stresses which in turn change the final shape of the component. Since the occurring phase transformations have a non-negligible influence on the mechanical material behaviour, the temperature evolution and the final shape of the component, the formed product phase fractions must be predicted accurately. Hence it is crucial to consider the effects of phase transformation during the numerical simulation of a hot forming process to obtain accurate results. An accurate prediction of the phase fraction evolution requires taking into account the specific cooling history of each material point, which can differ significantly from the path shown in the CCT diagram [6]. To this end various material models were developed in the past [12, 13, 14, 15, 16]. Selected models were used in numerical hot forming simulations by [3, 4, 17] to allow an accurate prediction of the temperature and fraction of each phase influencing the mechanical behaviour and the final shape of the specimen. The complete description of the transformation behaviour enables a prediction of the material properties as a result of the developed volume fraction of different phases.

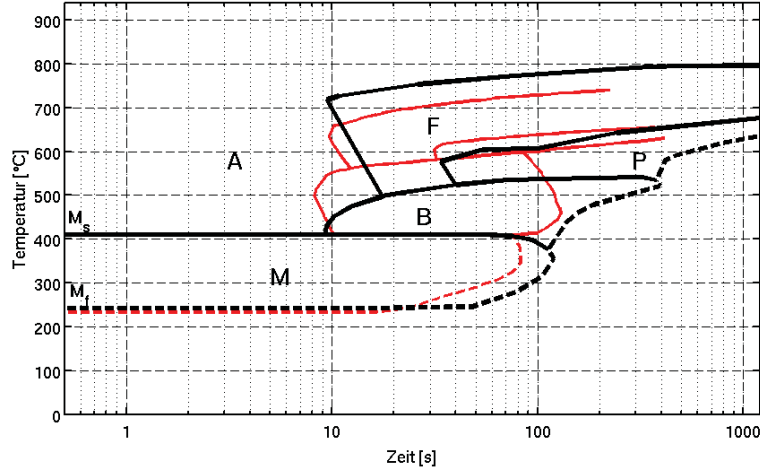


Figure 2.5: Shift of the phase regions in the continuous cooling diagram of a 22MnB5 press hardenable steel depending on preforming. [6]

2.3 Transformation Plasticity

Leblond [13] distinguishes between classical plasticity as a response of the material to variations of stresses or temperature and transformation plasticity as a response of the material to variations of the proportion of the phases. Transformation plasticity is induced by the volume difference between austenite γ with its face centered cubic *fcc* structure and ferrite, perlite and bainite α with a body centered cubic *bcc* structure and martensite α' with a body centered tetragonal *bct* structure. As published by [8] the changes in volume and shape of the crystals during the phase transformation enforce the softer phase to adapt to the environment of the harder phase. This means that because of the volume difference between the phases also microscopic internal stresses are generated in the phase which exhibits the lower yield stress. These internal stresses are sufficient to induce plasticity in the softer phase, even in the absence of any macroscopic external stress. As soon as two phases coexist microscopic plasticity is present in the weaker phase because of volume incompatibilities between the phases independently of whether the transformation proceeds or not. The so called Greenwood Johnson mechanism must therefore also influence classical plasticity. Hence plastic yielding can occur in the softer austenitic phase which has not yet transformed even if the applied stresses are smaller than the yield stress of austenite. Externally applied stresses can either initiate or amplify this effect.

Figure 2.6 shows the influence of the TRIP phenomenon on an austenitic wire loaded by a dead weight during cooling and martensitic transformation. Even

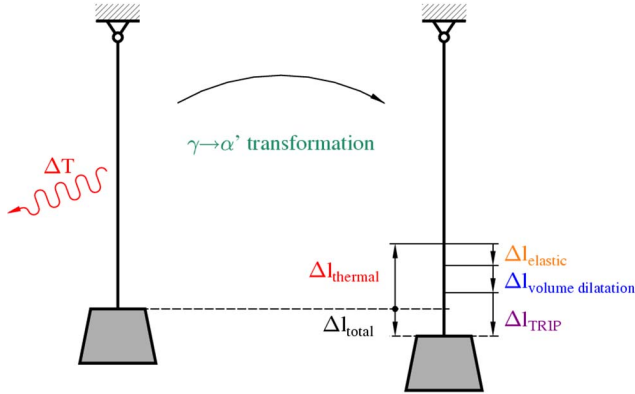


Figure 2.6: Additional elongation induced in a loaded wire during austenite- martensite phase transformation by Transformation Induced Plasticity (TRIP). [9]

for loads well below the yield limit of the material an inelastic strain contribution remains after such a cooling process. An external load leads to an elastic elongation $\Delta l_{elastic}$, the volume increase from austenite to martensite due to the different atomic packing density is considered as $\Delta l_{volume\ dilatation}$ and the thermal shrinkage during cooling is given as $\Delta l_{thermal}$. As can be seen in figure 2.6 the total elongation Δl_{total} is greater than the sum of the above mentioned contributions. The additional elongation is caused by the transformation induced plasticity effect abbreviated with *TRIP*. The total elongation is therefore given as

$$\Delta l_{tot} = \Delta l_{elastic} + \Delta l_{volume\ dilatation} + \Delta l_{TRIP} + \Delta l_{thermal}$$

The effect of the phase transformation as well as the TRIP effect are also visible in dilatometer curves of one entire heating and cooling cycle including phase transformations. As shown in figure 2.7 the linear change of strain during cooling is interrupted when the phase transformation starts and the strain is drastically increasing marked by ϵ^{tv} . The distance of the start and the end point can be explained by the TRIP effect named ϵ^{TRIP} .

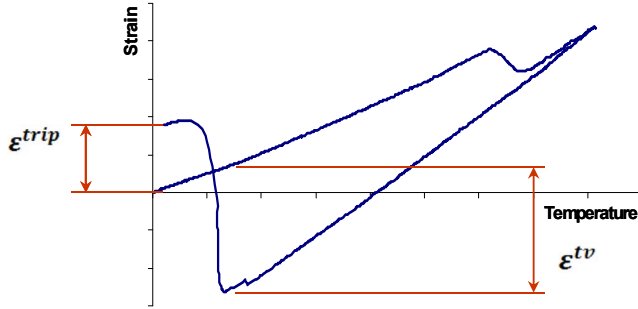


Figure 2.7: Remaining strain due to Transformation Induced Plasticity after one entire heating and cooling cycle including phase transformations.

In the case an applied stress is superimposed it orients these microscopic plastic deformations and this results in a net macroscopic strain in the direction of the applied stress. The so called Magee mechanism states that the transformation plasticity is due to an orientation of the newly formed phase by the applied stress. This mechanism is particularly pronounced during the formation of martensite, which develops in form of plates and therefore generates high shearing in the austenite. When no external load is applied, the plates are generally oriented randomly and therefore the macroscopic resultant of the microscopic stresses becomes negligible. An applied load generates internal stresses that favour certain directions of the formed martensite plates, which produces micro stresses with a non zero resultant. This generates transformation plasticity, which affects the overall shape of the specimen. The Magee mechanism operates only if the transformation proceeds and therefore it affects the response of the material to variations of the phase proportions but not to variations of the applied stress or temperature. [11, 24]

Since phase transformations induce an additional plastic behaviour during the hot forming process it is necessary to take it into account for the simulation of residual stresses in quenching operations.

2.4 Constitutive Models

There are in general two types of phase transformations depending on the cooling rate. At slower cooling rates the carbon atoms have enough time to diffuse to a new position in the newly formed atomic structure. Ferrite, pearlite and bainite can be formed by the so called diffusional phase transformation. At higher cooling rates the time is too short so that diffusion of carbon and other alloying el-

ement is inhibited and the non diffusional phase transformation occurs leading to martensite. The finite element simulation of the kinetics of phase transformations for steel requires mathematical models, which are based on physical and partly on empirical equations. Due to the different forming procedure, the diffusional and non diffusional phase transformation are described by separate constitutive models. [3]. Press hardening in particular aims to enforce the occurrence of the non diffusional austenite to martensite phase transformation.

2.4.1 Koistinen and Marburger Model

In case of press hardening the cooling rate in the quenching process is required to be above a critical value as visible in continuous cooling diagrams. Therefore the austenite to martensite phase transformation starts at a certain temperature designated as M_s temperature and proceeds only upon continuous cooling below this temperature. Martensite is characterized by a high mechanical strength which guarantees high resistance against further plastic deformation. That is the reason, why martensite as a structural constituent of steel makes it favourable for many technical applications. The kinetics of this phase transformation can be described by equation 2.1, which was first formulated by Koistinen and Marburger [12].

$$x_m = x_{\gamma_0} [1 - e^{-\alpha(M_s - T)}] \quad (2.1)$$

In equation 2.1, x_m represents the volume fraction of martensite, and x_{γ_0} is the initial volume fraction of austenite available for the reaction. x_m as well as x_{γ_0} vary between 0 and 1 for the pure austenitic and the pure martensitic phase respectively. The sum of the austenitic and martensitic phase follows $x_m + x_{\gamma_0} = 1$. M_s is the martensite start temperature and $(M_s - T)$ is the supercooling below the martensite start temperature. The constant α describes the velocity of the phase transformation. According to [12] $\alpha = 0.011$ for most steel types. It should be mentioned that the factor $\alpha = 0.011$ is based on an assumption that the temperature is 210 °C below M_s when 90% martensite is formed.

The equation developed by [12] describes the progress of transformation from austenite to martensite in carbon steels quantitatively. At any temperature below M_s a fraction of the austenite remaining will be transformed by a given additional temperature decrement. Obviously the formation of martensite is only dependent on temperature and not on time by using the model of Koistinen and Marburger. This reflects the diffusionless character of martensite formation. Since the Koistinen Marburger model is a purely temperature related formulation also the stress influences and additional strain impacts on the transformation behaviour are neglected. Moreover the load level does not have any influence on the martensitic start temperature by using the described model.

2.4.2 Leblond Model

The austenite to martensite phase transformation which takes place during the quenching step of the press hardening process causes the occurrence of Transformation Induced Plasticity (TRIP) in the weaker austenitic phase due to the volumetric difference between austenite and martensite. It is assumed that the TRIP behaviour has a non-negligible impact on the total strain state of the final component and hence the effect has to be considered in the numerical model as well. To this end the mathematical model proposed by Leblond [13] is used to account for the afore mentioned TRIP effect and its influence on the total strain during quenching.

Leblond suggested in his work a linear relationship between the resulting TRIP strain rate $\dot{\epsilon}_{ij}^{trip}$ and the stress deviator which is described by equation 2.2.

$$\dot{\epsilon}_{ij}^{trip} = \frac{3}{2} K_{tp} f'(\xi) \dot{\xi} s_{ij} \quad (2.2)$$

$$f(\xi) = (2 - \xi)\xi \quad \dot{f}(\xi) = 2(1 - \xi) \quad (2.3)$$

ξ is the martensitic phase fraction satisfying $0 \leq \xi \leq 1$, $\dot{\xi}$ is the time derivative of ξ , K_{tp} is the Greenwood Johnson parameter, $f(\xi)$ is the saturation function and $\dot{f}(\xi)$ its time derivative. The mathematical expressions for $f(\xi)$ and $\dot{f}(\xi)$ are given in equation 2.3. Leblond's model does not distinguish between the Greenwood Johnson effect [8] caused by the volumetric differences of austenite and martensite and the Magee effect [24] which is induced by an additional macroscopic external loading. It is also important to note that deformation prior to phase transformation is known to have an impact on the phase transformation kinetics and the fraction of the formed phases. The influence of the plastic deformation on the phase transformation behaviour is also observed for the austenite to martensite transformation. One explanation is that plastic straining of the austenite increases the number of nucleation sites in the metal structure which causes a shift of the phase regions in the continuous cooling diagram as explained in figure 2.5. In the present work, the effects of stress and strain on the martensite phase transformation are not yet accounted for. A thorough discussion regarding the effect of stress and strain on the bainitic and martensitic transformations can be found in Bhadeshia [25]. This type of effects will be included in a forthcoming work

2.5 Corrosion protection

Press-hardened parts used in car underbodies are often exposed to corrosive elements which makes coating of the material absolutely necessary. In general there

are coatings which offer just a barrier corrosion protection, by avoiding the direct contact of the corrosive component to the metal, or galvanic corrosion protection by corroding the coating element instead of the specimens material. This means that the coating acts as sacrificial layer and prevents the Fe substrate to be corroded. The advantage of a galvanic corrosion protection is that even in the case of a locally damaged coating the corrosion protection is still present by the surrounding coating. Nowadays zinc based coatings are often used since it provides galvanic corrosion resistance in service. The coating is usually applied by continuous hot dip galvanizing of the entire coil by running the steel sheet through a Zn bath held above about 420 °C, and then cooled to solidify the Zn. A relatively pure Zn coating with high cathodic corrosion resistance and a uniform coating thickness is obtained.

Since the coating is applied before the actual press hardening process starts, the coating experiences the heating, forming as well as quenching cycle of the press hardening process. For the quality of the manufactured component it is important to ensure that the coating can withstand the procedure and still fulfills its purpose in service. Pure zinc has, however, a much lower melting point compared to the substrate material, which means that the coating becomes liquid during austenitization in the oven. One way to avoid or mitigate the melting of the coating is a subsequent heat treatment to allow diffusion of iron atoms into the zinc coating. To this end the sheets are annealed after hot dip galvanizing. During galvannealing the sheets are heated to allow diffusion of zinc and iron atoms across the coating-substrate interface. The alloying of the zinc coating by diffused iron atoms increases the melting point of the coating. The resulting alloyed coating contains up to about 10 wt pct Fe, and is not as soft as a typical galvanized coatings. Near the interface between the Zn coating layer and the steel matrix, the concentration of Fe is high enough to form a solid solution of Zn in the ferrite. This solid solution phase has a high melting temperature. For example, at 30 mass pct of Fe in Zn, the melting temperature is approximately 950 °C [19]. For this reason galvannealed coatings are preferred over galvanized coatings. Nevertheless it must be considered that due to the diffusion of iron atoms during galvannealing the electrical potential is increased which means that the corrosion protection capabilities compared to the pure zinc coating are reduced.

The galvannealed coating contains in contrast to the galvanized coating already FeZn phases with a higher melting point at the beginning of the press hardening process. During austenitization the diffusion of iron atoms into the zinc coating as well as the diffusion of zinc atoms across the substrate-coating interface proceeds. At the end of austenitization the coating thickness is approximately twice as thick as before the heat treatment as depicted in figure 2.8 As can be also seen in figure 2.9 the homogeneous pure zinc coating contains now iron zinc phases with locally different zinc content and the surface is roughened. Although the Zn coating cannot provide full barrier coating protection after hot stamping due to the slight embrittlement of the α -Fe (Zn) layer, the coating can still provide ad-

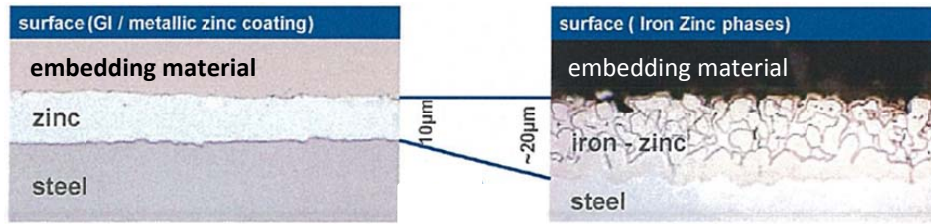


Figure 2.8: Evolution of the zinc coating during press hardening [22].

equate galvanic protection. However, the coating will not offer the same level of cathodic protection as the one available in the original state, i.e., in the galvanized or galvanized state, because the higher content of Fe in the Fe-Zn solid solution will increase the electrochemical potential of the coating [19]. Even though melting can be reduced by prior heat treatment, the difference in the thermal expansion coefficient between sheet and coating material cannot be overcome. For that reason periodically distributed thermal cracks can appear along the sample surface after quenching even in absence of any forming. These hairline cracks however end at the coating-substrate interface. During hot-stamping, however,

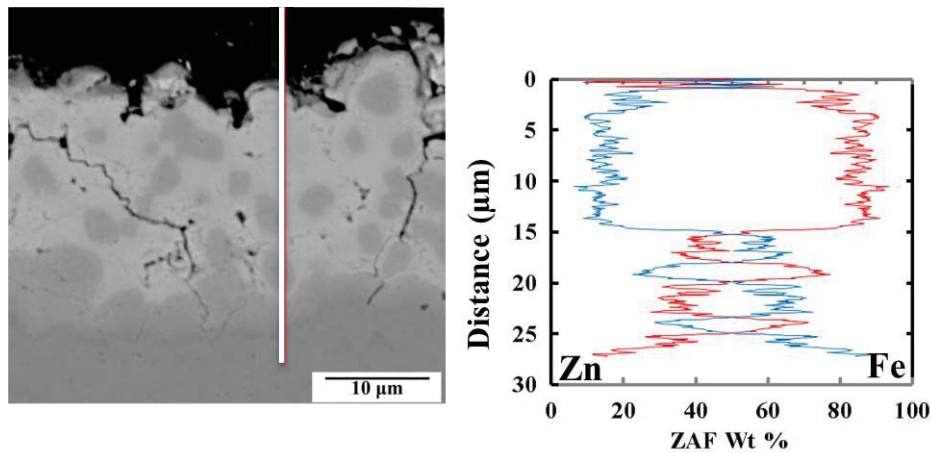


Figure 2.9: Micrograph and measured zinc and iron content of the coating after the oven dwell time [18].

cracks can form in the Zn coating and, under some circumstances, propagate into the steel substrate. The decrease in ductility can sometimes be so drastic that the ultimate tensile strength is not reached, and thus a reduced stress at failure may in some cases be the consequence [21]. The phenomenon is called Liquid Metal Embrittlement, abbreviated as LME.

2.6 Liquid Metal Embrittlement

LME is described as the reduction of total elongation to failure of a material, often a ductile metal, when it comes into contact with a liquid metal [17]. It is assumed to occur if a susceptible solid metal, a liquid metal and a critical stress are present at the same time. During hot-stamping of zinc coated sheet, all three of these basic criteria are met since the zinc coating becomes liquid and it is in direct contact to the solid and LME susceptible steel substrate. The coated sheet is hot formed which adds the required tension stress state and thus it is conceivable that LME may occur. Nevertheless, the actual conditions leading to LME are neither completely clarified nor quantified. It is still under investigation if zinc penetration occurs even before deformation by diffusion along grain boundaries or if the zinc is transported by surface wetting along already existing microcracks.

By identifying the conditions under which liquid metal induced cracking occurs, they may be avoided in the future during hot-stamping and the manufacturability of zinc coated press hardened parts may be enhanced.

Figure 2.10 shows the iron zinc phase diagram. It can be seen that the melting temperature of the iron zinc alloy decreases with increasing zinc content. The gray colored area shows the region where liquid metal induced cracks are likely to occur during the press hardening process. The upper border is chosen to be 950°C which is the maximum temperature during austenitization. The lower temperature border is dependent on the zinc content since the melting point decreases with increasing zinc content. At very low zinc contents LME cracks are avoided since the melting temperature of the alloy is even higher than the maximum austenitization temperature. The main aim is therefore to find a convenient forming temperature dependent on the coating's zinc and iron content to avoid liquid zinc phases during forming and at the same time to maintain a zinc content high enough to ensure cathodic corrosion protection.

This is not an easy task since higher coating thicknesses and zinc contents as well as shorter oven dwell times are desired to improve corrosion protection and to ensure cost effective manufacturing, respectively. Furthermore according to [21] a minimum temperature is required for the complete austenitization of the substrate material, a maximum temperature is given to avoid evaporation and oxidation of the coating and a maximum dwell time should not be exceeded to prevent the corrosion protection capabilities.

In the case liquid zinc phases are present during forming there is a high risk of crack formation and propagation into the steel substrate. According to [19] the LME cracks are initiated at the solid-liquid metal interface. During the annealing prior to hot stamping, the Fe-Zn intermetallic compounds are transformed into a Zn-rich liquid phase. Grain boundaries in α -Fe (Zn) appear to be already wetted by liquid Zn before tensile loading [19]. This grain boundary film leads to an em-

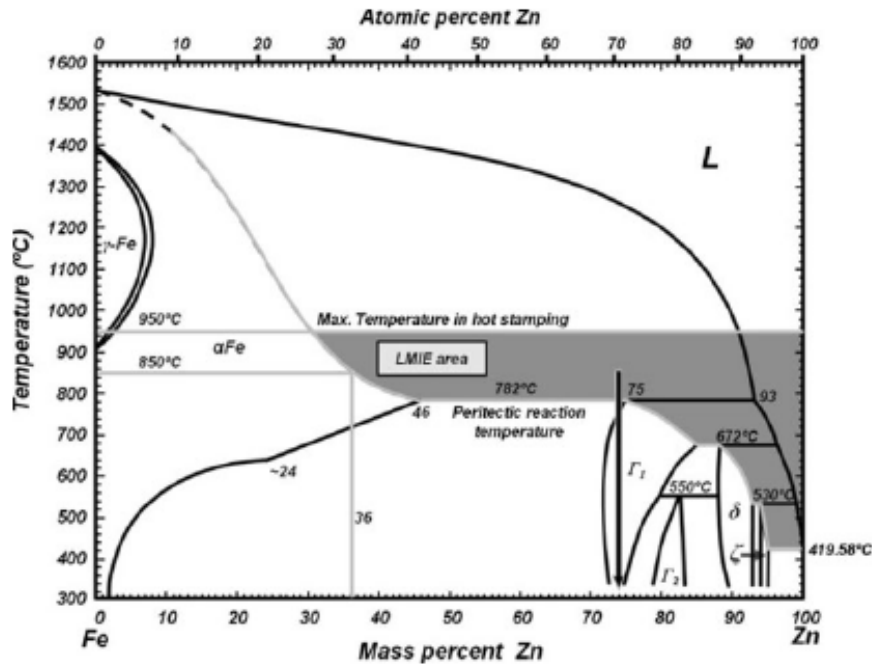


Figure 2.10: Iron - zinc equilibrium phase diagram with LME critical area colored in gray [19].

brittlement of the substrate material and grain boundary decohesion takes place. When sufficient stress is applied to achieve the required plastic deformation, the Zn-diffused boundaries act as crack initiation sites [19]. During tensile loading, the loss of grain boundary cohesive strength of α -Fe (Zn), due to the presence of a liquid metal leads to crack formation in the coating and propagation of the crack into the steel substrate. After the LME cracking of the α -Fe (Zn) layer, the liquid Zn continues to penetrate along the austenite grain boundaries of the austenitic steel matrix. Figure 2.11 shows schematically the penetration of liquid zinc along the austenitic grain boundary and the crack propagation as soon as a critical tensile stress is applied. As the cracks propagate with a high velocity (1 ms⁻¹), the ductility and toughness of the material are drastically decreased [19]. Zn penetration below the alloyed layer into the substrate was not observed in the undeformed material under any conditions. These data suggest that deformation at elevated temperature was also required for Zn penetration to occur into the substrate. Figure 2.12 shows a microscopy image of a LME crack detected by [18]. The zinc visible at the crack tip appears brighter than the substrate in backscatter mode. The bright regions indicate the zinc penetration into the substrate. This kind of zinc penetration is, however, only observed in deformed samples and could not be detected in undeformed samples.

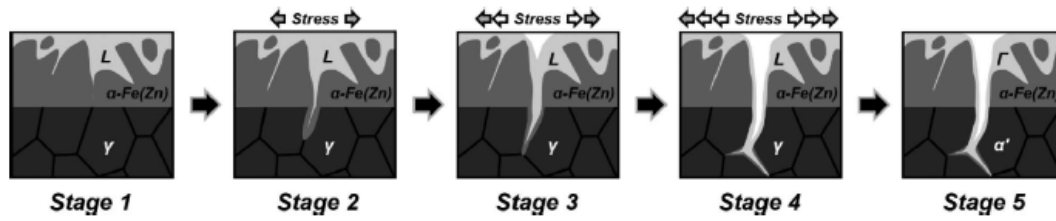


Figure 2.11: Possible LME crack formation mechanism [23].

Figure 2.13 shows zinc coating cracks which usually stop at the coating-substrate interface but could also propagate into the steel substrate if LME conditions are met.

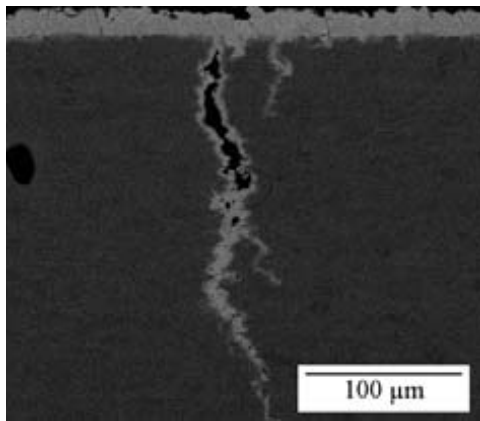


Figure 2.12: Liquid zinc at crack tip in the steel substrate [18].

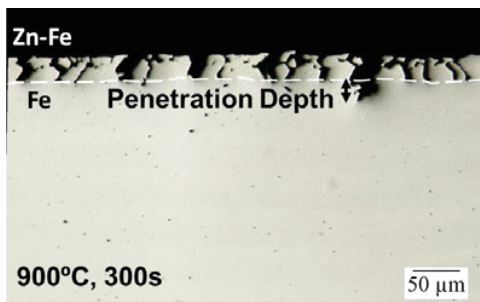


Figure 2.13: Surface cracks in the zinc coating and propagation in the substrate at one position [18]

In prior works by [18, 19, 21] different approaches of how LME can be avoided have been published. [19] suggested that LME can also be avoided if the deformation stress is lower than a “critical stress” parameter which was unique for each combination of temperature, hold time, and strain rate. [19] mentioned that if enough diffusion between the coating and substrate takes place in a manner that significant amounts of liquid are not present in the coating at this temperature, LME could potentially be avoided under this condition. The authors proposed

that with increased hold time, Fe-Zn intermetallics have time to form at the steel surface and thus prevent further contact of the Fe and Zn and subsequent embrittlement due to liquid Zn. According to [19] LMIE can also be avoided by deformation below the melting temperature of the Fe-Zn intermetallics, but this approach leads to the transformation of the austenite to strain-induced ferrite and bainite, and it results in a considerable reduction of the strength of the hot-press-formed part. Additionally [21] investigated the influence of the oven dwell time, forming temperature and coating thickness on the crack formation behaviour. It was found that an increasing dwell time decreased the crack depth but at the same time increased the number of cracks. This could be explained by a higher iron content in the coating due to the improved diffusion during longer oven dwell times and therefore an enhanced melting temperature of the coating. A lower forming temperature decreased the crack depth since less liquid zinc phases were present to induce the crack. Lower coating thicknesses decreased the crack depth as well due to shorter diffusion ways of the iron atoms in a thinner coating. With increased soak time (300 s and 600 s) the amount of α (bcc Fe-Zn containing about 35 wt pct Zn) phase increased and the Zn-rich Γ_1 regions (about 70 wt pct Zn) decreased [18, 21].

Figure 2.14 shows schematically one symmetry half of the tools and sheet during hot forming.

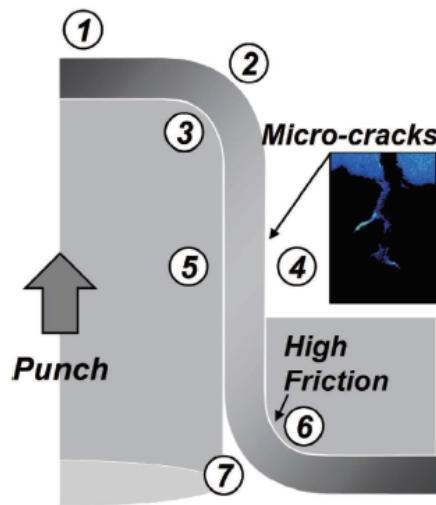


Figure 2.14: Schematical description of the hot sheet forming of a hat shaped profile [23]

As the punch is moving up the sheet is drawn and bent into the die. Due to drawing and bending stress is induced especially in the surface near region. At the same time high friction is present specifically between die radius and the outer

side of the sheet surface since the contact forces are highest in the region marked by 6. When analysing the bending in detail it can be seen that on the inner sheet surface the forming history in the region marked by 7 is tension and subsequent compression during rebending. At the position 6 it is vice versa since the material is first compressed running along the radius and is subsequently stretched during rebending.

The forming history on the outer and inner sheet surface and its influence on the crack behaviour is depicted in figure 2.15. Cracks are assumed to be deeper on the outer side due to tension and friction.

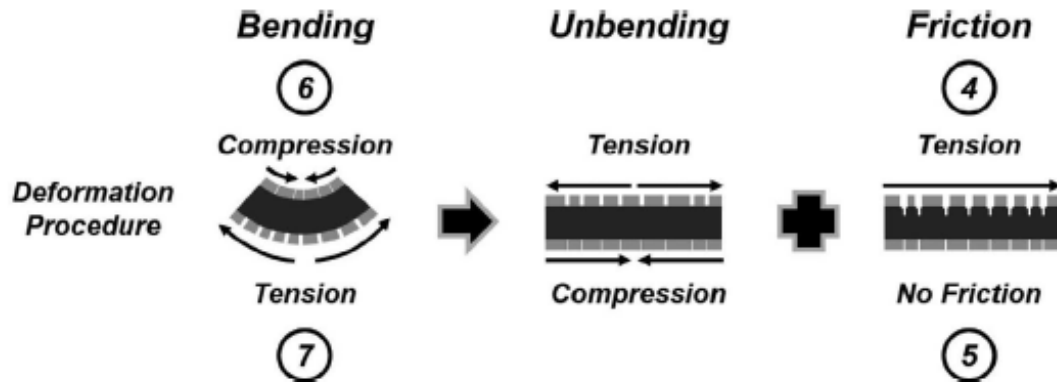


Figure 2.15: Representation of the forming history of points 4, 5, 6 and 7 of figure 2.14 and its influence on the crack formation [23]

Chapter 3

Experiments

3.1 Cooling experiments

To obtain the initial forming temperature and to investigate the cooling behaviour during transfer from the oven to the press, required as input data for the numerical simulation, cooling experiments with rectangular press hardenable steel sheets were conducted. A thermocouple was attached centered on the upper side of the sheet which allowed measuring the temperature evolution during oven dwell time and transfer to the press. During cooling the temperature was additionally measured by pyrometers. Figure 3.1 shows the difference between the thermocouple and pyrometer measurements. It can be seen that the temperature measured by the thermocouple is slightly higher than the pyrometer measurement. Even though the difference is not large it has to be considered, since it is absolutely crucial to predict the start of the phase transformation in the subsequent forming simulation accurately. It is not possible to measure the temperature by thermocouples during the forming experiments described in section 4. Therefore the temperature of the sheet during the transfer to the press is measured by the same pyrometers used during the cooling experiments and has to be adapted by adding the known deviation of the pyrometer and thermocouple measurements. The time of transfer varies according to the desired forming start temperature. By means of these experiments the exact temperature at the beginning of forming could be obtained as input parameter for the simulation.

The cooling process was then simulated in a finite element heat transfer analysis. Due to the symmetry only one quarter of the sheet had to be modeled. The temperature dependent heat transfer coefficients of free convection were calculated by means of dimensionless numbers, in particular the Nusselt and the Rayleigh number. The emissivity value was set constant throughout the cooling simulation according to the emissivity setting of the pyrometer during the cooling experi-

ments. The previously measured cooling curves were used to validate the chosen input parameters of free convection and radiation used in the numerical model. The measured and simulated temperature evolutions during cooling were compared to each other. The results showed already good agreement to each other as seen in figure 3.2.

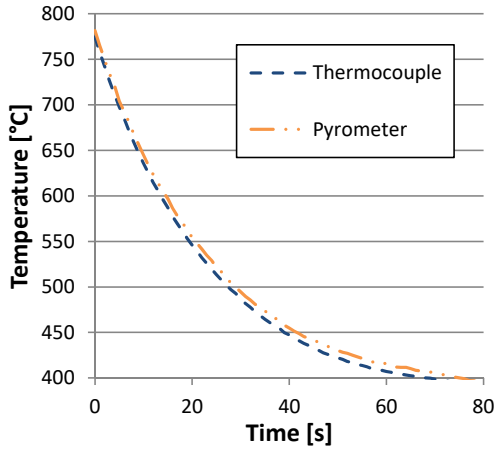


Figure 3.1: Comparison of the thermocouple and pyrometer measurements during cooling under atmospheric conditions [28].

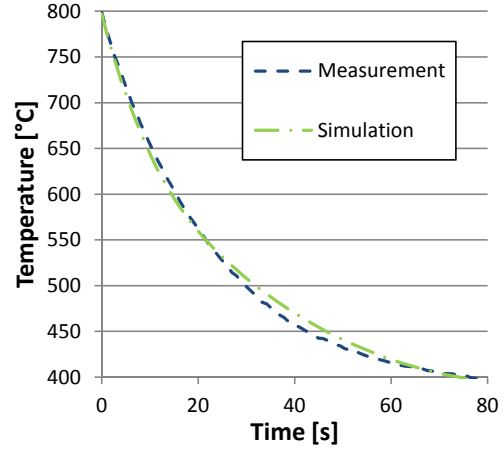


Figure 3.2: Comparison of measured and simulated temperature evolution during cooling under atmospheric conditions [28].

3.2 Forming experiments

Forming experiments of a simple hat shaped profile as depicted in figure 3.3 were conducted to allow a validation of the numerical forming and press hardening model by comparing the final shape and wall thickness distribution. During the experiment a press-hardenable steel sheet was austenitized in the oven. After complete austenitization a robot removed the steel sheet from the oven and transferred it to the die. During this process the sheet further cooled down due to radiation and convection. Shortly before the sheet was placed into the die, the temperature was measured by pyrometers. High tool velocities were required to ensure forming to final shape in one stroke in a still austenitic state. After forming the cooled die remained closed until completion of the martensitic phase transformation in the specimen. After removing the specimen from the die it cooled down to room temperature at atmospheric conditions and the wall-thickness was measured at different positions along the cross section. Three different forming

start temperatures were chosen to study their influence on the phase transformation and the final specimen shape.



Figure 3.3: Press hardened hat shaped profile formed during experiments

Chapter 4

Simulation

4.1 Press Hardening Simulation

The simulations were performed using the commercial software package Abaqus/Standard. According to [3, 4] a strongly coupled temperature-displacement analysis is required due to the fact that contact between the die and the sheet during forming and press hardening significantly influences the temperature evolution. Figure 4.1 shows the press hardening process steps after complete austenitization in the oven. The austenitized sheet is considered as the stress free initial state. The black rectangle in figure 4.1 marks the transfer, forming and quenching steps including phase transformation considered in the simulation.

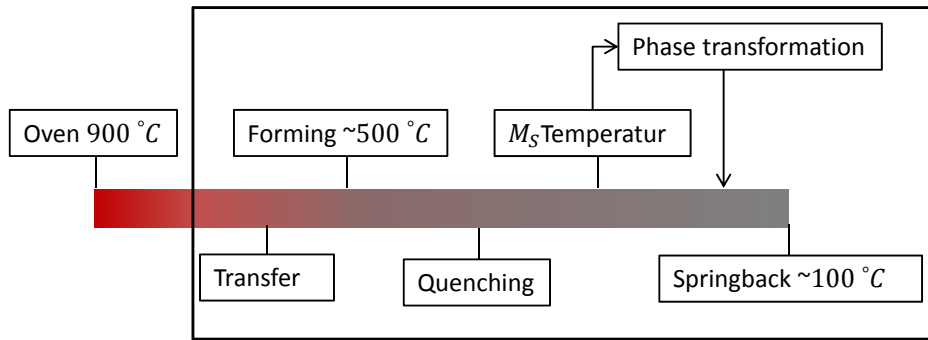


Figure 4.1: Schematic representation of the press hardening process steps after complete austenitization, considered in the simulation.

Due to the shape of the formed profile a 2D plane strain model representing the cross section of the hat profile sufficed to study the principle thermo-mechanical response. To reduce the calculation time only one half of the symmetric hat

profile was simulated. Since the heat flux from the sheet to the die during forming and especially quenching must not be neglected all tools were modeled as elastically deformable parts with temperature degree of freedom and an initial temperature of 25°C. The model contains three tool parts, the die, the punch and the blank holder, see Figure 4.2. The tool's thickness was chosen such as plastic deformation is avoided and the temperature increase is only restricted to the sheet contact region.

For the material description the flow curves were available at various temperatures within the relevant interval from room temperature to temperatures just above austenitization. For implementation into Abaqus/Standard classical Mises plasticity with isotropic hardening was chosen. Since the temperature range of the press hardening process is significant, it is also crucial to provide all material properties like Youngs modulus, thermal conductance, specific heat, thermal expansion etc. dependent on the temperature. Also the interaction properties, i.e. the friction coefficient as well as the heat transfer coefficient across the contacting bodies are temperature dependent as investigated by [6, 26]. Note that the latter is also a function of the contacting pressure. This effect had to be taken into account in the model.

The press hardening simulation was divided into three steps. During the first step the sheet with an initial temperature between 500°C and 700°C was formed in one single stroke. The initial forming temperature was previously determined by means of the transfer simulation and experiments as explained in section 3.1. The press hardening simulations accounts for all sources of non-linearities, i.e. large displacements, material non-linearities, as well as contact and friction. The process parameters such as drawing depth and tool velocity are set in accordance to the experimental process parameters. In the second step the tools remain closed for 5 seconds and the sheet cools down due to the heat flux to the die. In the third step the tool contact is deactivated and the specimen cools down to room temperature under atmospheric conditions.

The two dimensional solid elements CPE4RT with four nodes, displacement and temperature degree of freedom and reduced integration were used for the tools as well as the specimen. A fine mesh with an element length of 0.5 mm and 10 elements evenly distributed along the sheet thickness was used for the specimen to enable smooth bending. A coarser mesh was used for the tools with the exception of the tool radii. A finer mesh was applied to the curved tool contours to ensure a smooth radius and to avoid unrealistic forming conditions due to the element discretization as can be seen in figure 4.3.

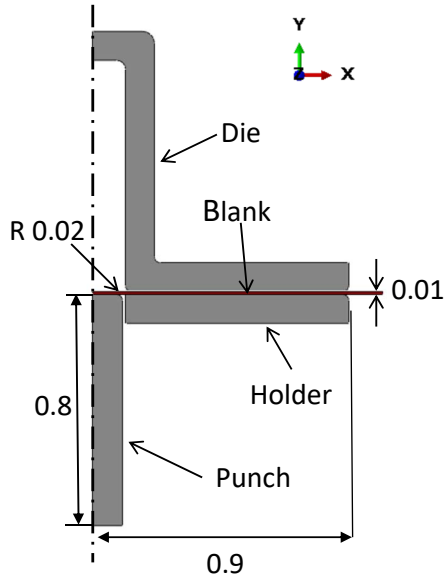


Figure 4.2: Positions of tools and specimen before forming. Dimensions are normalized by the plate length [28].

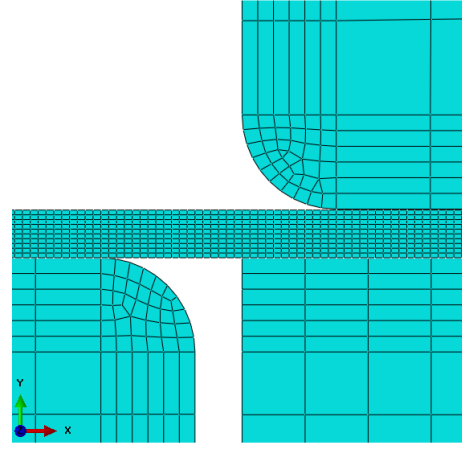


Figure 4.3: Detail of the sheet and tool mesh located at the tool radii

In order to account for the phase transformation during quenching and its influence on the final shape and mechanical properties of the formed component, an accurate thermo-mechanical-metallurgical model has to be provided. The model considers the interaction between the mechanical field induced for example by forming and thermal expansion, the thermal field evoked by phase transformation, plastic forming and friction, as well as the microstructure evolution in dependence on the temperature and the stress-strain state. Moreover the influence of the microstructure on the mechanical and thermal properties of the material is considered. The model's functionality is adapted to these particular analysis requirements by taking advantage of various subroutines available in Abaqus/Standard [28].

Figure 4.4 explains the interactions of the mechanical and thermal field considered by the strongly thermo mechanically coupled model as well as the microstructure evolution included by means of Abaqus/Standard user subroutines.

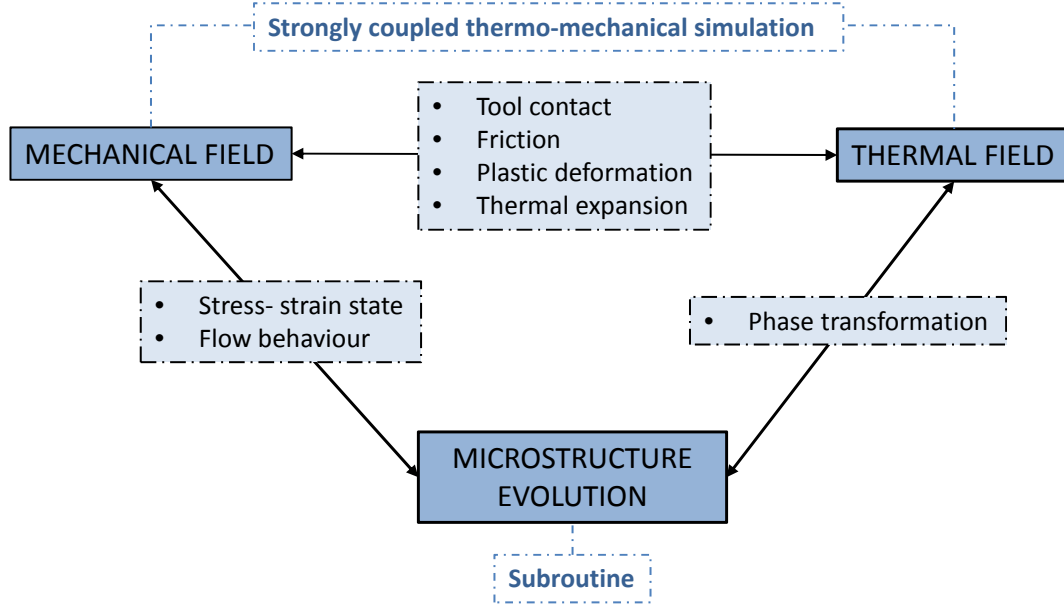


Figure 4.4: Schematic representation of the numerical press hardening model and its interactions.

The user subroutine USDFLD was used to calculate the phase fraction of martensite $\zeta_M(T)$ following a slightly modified kinetic law of Koistinen and Marburger used by [3, 27]. If the simulated temperature is below the martensite start temperature M_s and the cooling rates are high enough, martensitic transformation occurs according to

$$\zeta_M(T) = 1 - e^{-\alpha(M_s - T)^n}$$

where T denotes the current temperature and α as well as n are material parameters.

In this specific case the martensitic phase fraction is stored as a field variable. The material properties are then made dependent on this field variable. The field variable along with some other state variables, updated in the USDFLD routine, are passed into the user subroutines UEXPAN, CREEP and HETVAL, which are also called at the integration points. Subroutine UEXPAN is used to define the incremental thermal strains as function of temperature and state variables representing the phase contents. In this way the shrinking during cooling as well as the volume change due to the phase transformation are considered. State variables updated by UEXPAN are passed into the user subroutine CREEP which accounts for transformation induced plasticity (TRIP) effect. The austenite to martensite phase transformation influences also the temperature evolution due to the latent heat release during phase change. To this end the values of state

variables, already updated by USDFLD, UEXPAN and CREEP subroutine, are finally passed into the HETVAL subroutine, to define the heat flux due to internal heat generation in the material as it occurs during the phase change [28]. A comprehensive description of the interplay of the subroutines necessary for capturing the effects of martensitic phase transformation and implementation details can be found in [10].

Chapter 5

Results

The simulation results were compared to experimental results to validate the chosen input parameter. In a first step the wall thickness of the specimens formed during the experiments were measured at five different positions distributed along the specimen's half cross section. In a second step the flange width of the hat shaped profile was measured. The positions of the wall thickness measurement as well as the location of the flange width measurement are shown in figure 5.1.

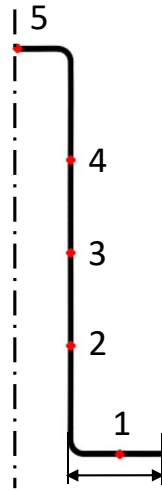


Figure 5.1: Sheet thickness measurement and flange width measurement locations [28].

Due to the symmetry only one half was simulated and compared to the experimental results. As it can be seen in table 5.1 the wall thickness as well as the flange width predictions already show good accordance to the measurements.

This is an indication that the mechanical forming behaviour as well as the forming temperature and friction between the tool and the sheet have been chosen correctly.

Table 5.1: Wall thickness and flange width of simulation and experiment for three forming temperatures

Position	1	2	3	4	5	<i>Flange width</i>	<i>Temperature</i>
Simulation	1.52	1.38	1.37	1.38	1.52	28.0 mm	500°C
Experiment	1.53	1.39	1.38	1.38	1.50	28.9 mm	500°C
Simulation	1.51	1.38	1.38	1.38	1.50	27.9 mm	580°C
Experiment	1.52	1.39	1.40	1.39	1.49	28.6 mm	580°C
Simulation	1.51	1.38	1.38	1.37	1.49	27.6 mm	670°C
Experiment	1.51	1.40	1.39	1.41	1.48	27.8 mm	670°C

Since the simulation results for the wall thickness and flange width already showed satisfying agreement with the experimental evidence, the press force which was recorded during the forming experiment has been compared to the simulated tool's reaction force to further investigate the reliability of the simulation. A validation of the required press forces is important since it also indicates that the plastic forming behaviour, specimen's temperature and friction have been chosen correctly as input parameter for the numerical model. Since it is challenging to predict the exact conditions during forming, especially regarding the friction coefficient and the thermal conductance in dependence of surface clearance and contact pressure, four variants were simulated using two different friction coefficients and two sets of thermal conductance values whereby letter L indicates the lower and letter H the higher values of the varied parameters.

The simulated as well as measured press force during forming is depicted in figure 5.2.

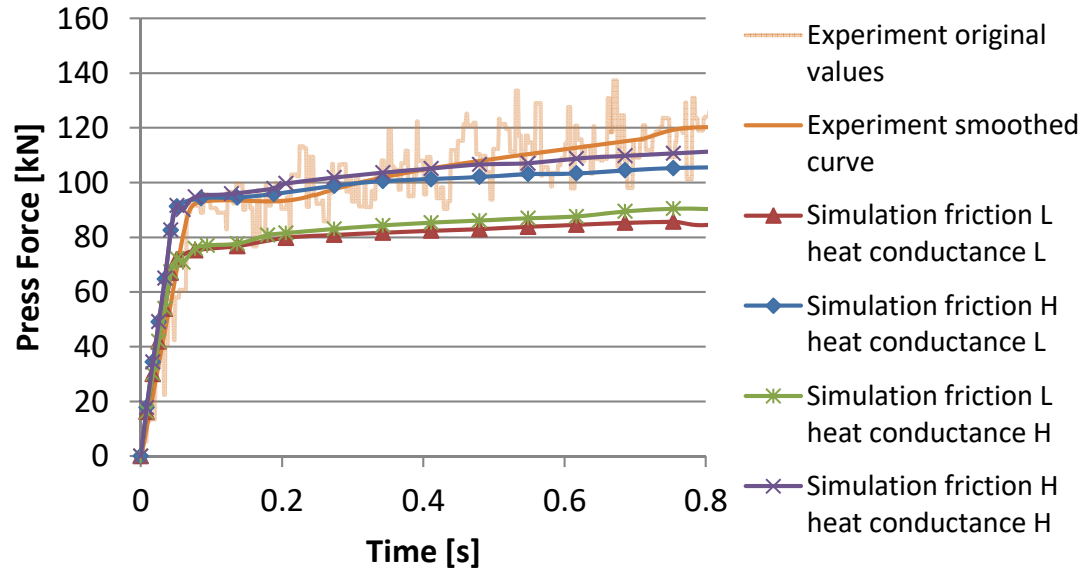


Figure 5.2: Comparison of measured and simulated press force during forming with lower L and higher H set of contact parameters [28].

According to figure 5.2 the higher friction coefficient and higher thermal conductance between tools and sheet leads to a press force closest to the measured press force during forming.

After validation of the material model used in the press hardening simulation a detailed investigation of the stresses and contact conditions was conducted.

Figure 5.3 shows the nominal stress in the element's longitudinal direction and the present contact areas at the beginning of the forming step. It can be seen that high tensile stresses are induced due to bending on the outer fiber of the sheet while compressive stresses are caused at the inner fiber of the sheet closer to the tool radius. The contact areas between sheet and tools are small compared to the total tool surface available. This means that contact forces are applied very locally on the sheet but nevertheless the sheet is still able to slip through the drawing gap.

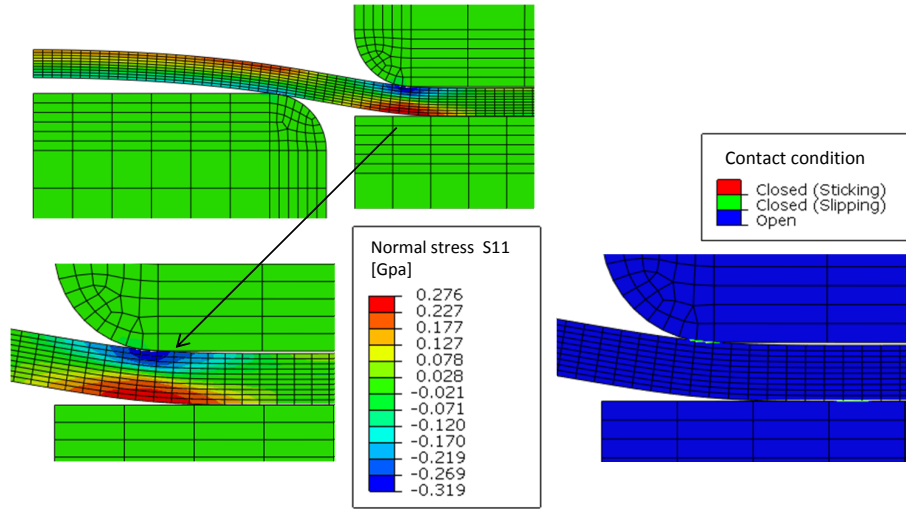


Figure 5.3: Normal stress (left) and contact conditions (right) of the sheet at the forming start.

Along with the forming progress the tensile as well as the compressive stresses increase and the area in contact to the tool is enhanced as seen in figure 5.5 on the left and right hand side respectively.

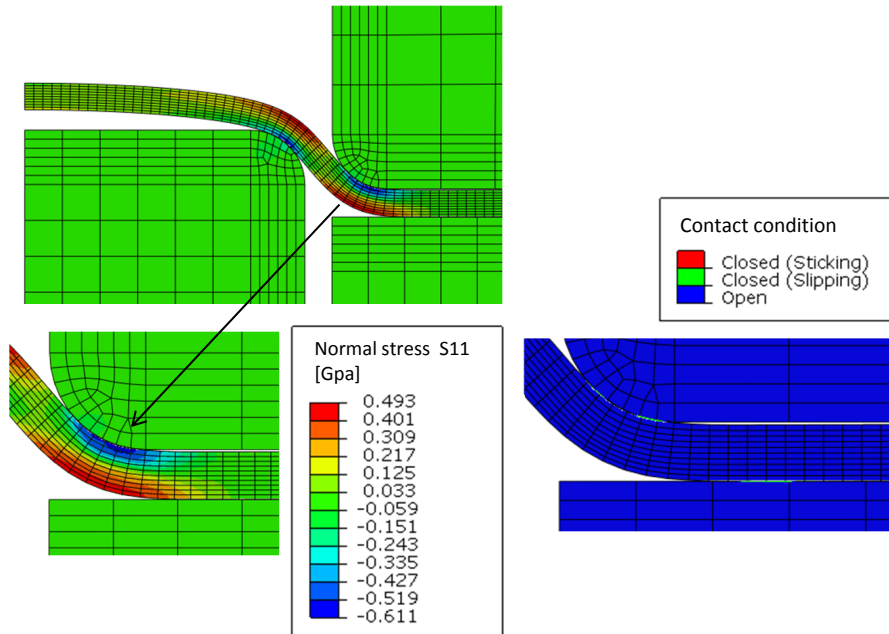


Figure 5.4: Normal stress (left) and contact conditions (right) after further forming progress.

Figure 5.5 shows the normal stresses and contact conditions present after further progress of the forming procedure. In that state the sheet is fully bended, enforced by the tool radius and afterwards completely rebended while slipping into the drawing gap. This induces tensile and subsequent compressive stresses in the outer fiber and compressive stresses followed by tensile stresses in the inner sheet fiber closer to the tool radius. The stresses increase continuously throughout the forming procedure since the sheet temperature is decreased due to the heat flux to the tool. This means that the formability of the sheet decreases towards the end of the forming step. As can be seen no slipping of the sheet surface in reference to the tool surface occurs in this state at the upper sheet radius.

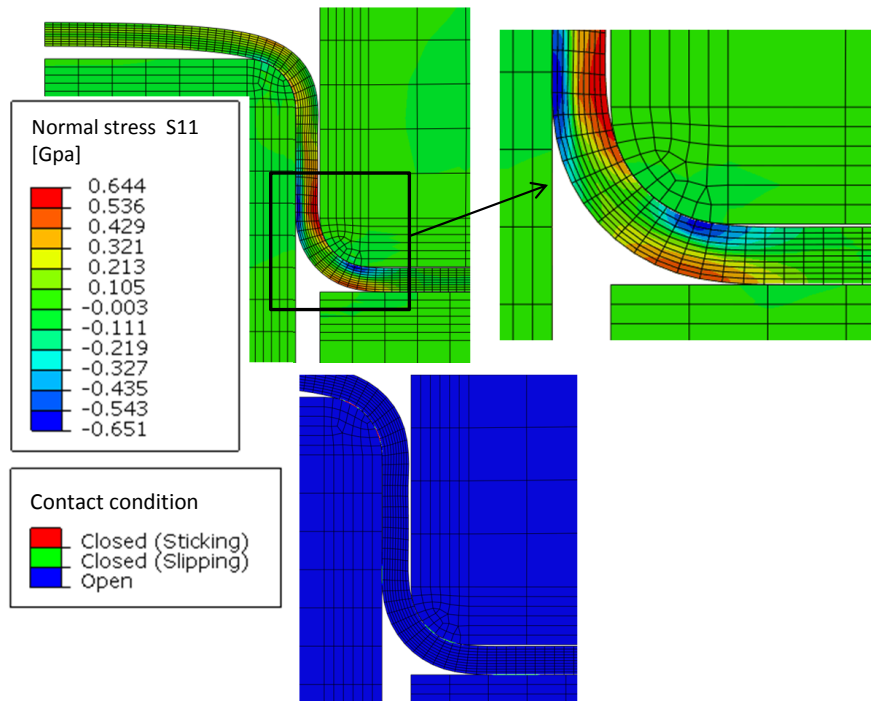


Figure 5.5: Normal stress (top) and contact conditions (bottom) when the sheet is completely drawn in the drawing gap.

Figure 5.6 and figure 5.7 represent qualitatively the normal stresses in longitudinal element direction of one location on the outer and inner fiber of the sheet respectively. The opposite occurrence of compressive and tensile stresses is recognizable.

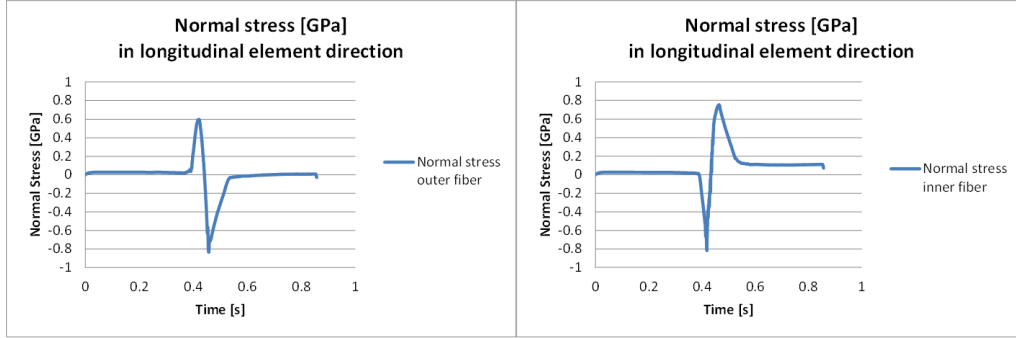


Figure 5.6: Representative normal stresses in the outer fiber during forming

Figure 5.7: Representative normal stresses in the inner fiber during forming

The forming history is known to have significant impact on the mechanical material behaviour and the phase transformation kinetics. These effects will be considered in forthcoming work.

Chapter 6

Conclusion and Outlook

For a deeper insight into the mechanisms leading to LME a strongly thermo-mechanically coupled simulation of the press hardening process of a simple hat shaped profile was developed. Simultaneously press hardening experiments of the same tool geometry were conducted.

Temperature dependent material and interaction parameters were required for the numerical analysis and to this end several experiments have been carried out in advance to obtain all necessary input data for the subsequent numerical analysis.

The numerical model comprises the forming and quenching step as well as the austenite to martensite phase transformation to reflect the press hardening process accurately. The wall thickness distribution along the specimen's cross section, the flange width and the press force were chosen as comparison factors, thus the used material, interaction and process parameters were successfully validated. The good accordance of the simulated and experimentally measured temperature evolution of the sheet during cooling under atmospheric conditions implied that the convection and radiation heat transfer coefficients have been chosen correctly. First investigations were conducted to analyse the stress state and contact conditions of the sheet during forming.

In future work the mechanisms and influencing factors of LME will be further investigated by means of the described numerical model by using the forming history i.e. the stresses, strains, contact pressures, and temperatures developing during the process in each point on the sheet's surface along the component's cross section. This allows to correlate the temperature, stress, contact conditions etc. at each location throughout the whole cycle to each other. Hence the points and time where critical conditions may occur, can be determined.

To validate this approach, micrographs of samples cut out from the prior press hardened components at different positions equally distributed along the compo-

ment's cross section will be prepared. Thus the regions showing cracks propagating in the steel substrate will be identified. A detailed numerical analysis will be subsequently done at positions where cracks propagate into the steel substrate according to the micrographs. Assumptions on the crack initiation and propagation mechanism will be made by using the evolution of the present stresses, temperatures and contact conditions at those specific locations during the press hardening process.

This will help to evaluate if the cracks are initiated already during forming or if the phase transformation to martensite is influencing the crack propagation as well. Furthermore it will be investigated if the cracks are formed instantaneously or if they propagate during the forming step.

By microscopic investigations it will be analyzed if the cracks propagate in an intercrystalline or transcrystalline manner and if zinc can be detected along grain boundaries or on the crack's surface. Cracks propagating in an intercrystalline manner indicate that possible grain boundary diffusion during the oven dwell time might cause the embrittlement and subsequent cracking of the specimen as soon as a critical tensile stress occurs during the press hardening process. Cracks propagating continuously instead of instantaneously indicate that surface wetting, in other words a zinc layer on the crack surface, will cause a slower step by step crack propagation. Finally the mechanisms leading to LME should be clarified. The findings of this work will eventually contribute to avoiding conditions critical to LME occurrence in the future.

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