ABSTRACT

The unique feature of thermomechanically treated martensitic steels is the deformation and the modification of the austenite and the inheritance of the generated microstructure to the product martensitic phase. The main factors controlling the desired microstructure prior to quenching are the deformation temperature, rate, and strain and the devolution of time and temperature between deformation and quenching. The correlation between the process parameters, microstructure and mechanical properties is described based on results for different spring steels. Compared to conventionally heat treated samples strain close to the onset of dynamic recrystallization of the austenite improves both, the strength and the ductility for 50CrV4 and 55SiCr6. Independent of impurity content, grain refinement of austenite followed by work hardening leads to superior properties for 55SiCr6. For thermomechanically treated samples no strong effect of austenitization temperature on ductility or toughness was observed for 36CrNiMo4 and 55SiCr6. Thermomechanically treated 50CrV4 and 55SiCr6 are not prone to embrittlement by phosphorous. Deformation of austenite influences the shape and distribution of carbides within the matrix and at the prior austenite grain boundaries.

It is concluded that utilization of TMT can be beneficial for increasing the ultimate tensile strength to levels above 2200 MPa and at the same time maintaining the ductility obtained at strength levels of 1500 MPa by conventional heat treatment. Additionally the endurance limit is increased and embrittlement does not occur.

KEYWORDS

Thermomechanical treatment, tempered martensitic embrittlement, austenite conditioning, carbide precipitation, dynamic recrystallization, recovery.

INTRODUCTION

Today, quenched and tempered, medium carbon martensitic steels are widely used for applications where high mechanical strength is the main design driver. But in most engineering applications beside strength a good ductility, toughness or fatigue and corrosion behavior is demanded. To obtain a good compromise between strength and toughness the as quenched martensite is reheated to temperatures between 150 and 700°C to obtain the required level of toughness without a big loss in strength. During the tempering the carbon in solid solution is rejected from the martensite. Depending on the tempering temperature and time various carbides with different shape and dispersion are precipitated and determine the strength and toughness. The strength increases with decreasing tempering temperature while the ductility or toughness decrease. Thus higher strength levels can not be achieved by conventional heat treatment methods that only offer a few possibilities to set the material properties.
Additionally, most commercial steels contain impurities that influence the toughness and ductility significantly. The well known embrittlement phenomena observed around 350°C is such an example where grain boundary segregation of impurity elements together with carbide films at grain boundaries deteriorate the mechanical properties of the material [1-5].

Research at our Institute has shown that the thermomechanical processing can result in improved properties of existing steels grades [6-10]. It has been shown that the properties of the tempered martensite can be controlled by a conditioning of the parent austenitic phase. By deforming the austenite, its grain size and grain substructure can be controlled in such a way, that a high strength martensitic steels with excellent ductility, toughness or fatigue limit is obtained. In this paper we describe the theoretical background of the thermomechanical treatment and present an optimization strategy for martensitic high strength steels. The objective of this paper is to show ways of optimizing the properties without any change in chemical composition. Therefore we present data for different steel alloys in order to show the benefit of the thermomechanical treatment for quenched and tempered martensitic steels in general.

PROCESS PARAMETER – MICROSTRUCTURE – PROPERTY

The final properties of any specific material, i.e. an alloy with fixed composition, are defined by its microstructure or defect structure. The microstructure itself is the result of the process parameters used during production. In this chapter the main process steps of quenched and tempered martensitic steels and their effects on microstructure and thus on the properties are discussed. The conventional heat treatment for producing high strength martensitic steels consists of austenitization, followed by quenching and tempering. Control of the microstructure in a thermomechanical treatment is carried out by deformation of the austenite prior to quenching. Therefore the changes induced by thermomechanical treatment are discussed within the section of austenite conditioning after the austenitization.
**AUSTENITIZATION**

**Solid solution.** One objective of the austenitization is to obtain a homogeneous solid solution of austenite, where all the carbon and alloying elements are statistically distributed. A homogeneous distribution of carbon and alloying elements is necessary to obtain uniform properties. The amount of carbon in solid solution determines the hardness of the final martensite [11, 12]. If metal carbides other than iron carbides are present a minimum austenitization temperature or time is needed to dissolve all the carbides and distribute the carbon. Such metal carbides can be compounds of carbon with microalloying elements such as vanadium. Their presence can lead to smaller grain size or to a bimodal grain size distribution [11,13]. Another problem which can occur during austenitization is equilibrium segregation of impurity elements such as phosphorous to the grain boundary. Phosphorous enrichment at grain boundaries can lead to brittle failure of the final part [14-17]. The equilibrium grain boundary concentration of impurity elements which have a low solubility in austenite depends on the bulk concentration and on the austenitization temperature and time [18, 19]. The higher the austenitization temperature the lower is the concentration of the impurity elements at the austenite grain boundaries [20]. Summarizing the above remarks it is necessary to use high austenitization temperature in order to obtain a uniform distribution of carbon and alloying elements and to minimize the enrichment of impurity elements at the grain boundaries. But as in most problems in material science, there is a contradiction to be solved. In contrast to the homogenization during austenitization the grain size refinement requires low temperatures. This is described below.

**Grain size.** The austenite grain size determines the toughness of high strength steels. Ohtani et. al. showed that the ductile to brittle transition temperature decreases with the average austenite grain size [21]. As described by Morris et. al. the main objective of grain refinement is to limit crystallographic coherence length for transgranular crack propagation [22]. The latter author defines an effective grain size which is the coherence length on {100} planes that determines the cleavage crack length. Because lath within a packet or block are in close crystallographic alignment, they behave as a single crystal of martensite. Hence, the {100} coherence length is defined by the packet size or block size of the final martensite. The packet size in turn is limited and depends on the austenite grain size [23, 24]. Thus small austenite grain size is
Fig. 4 Schematic presentation of possible austenite microstructures (blue circles) after a deformation prior to quenching

desirable to obtain high toughness for a specific strength level. Grain refinement further leads to increase of the grain boundary area. Thus for a given impurity concentration more segregation sites are offered to the same amount of atoms leading to a lower grain boundary concentration of the atoms at the grain boundaries.

For the same initial microstructure and constant heating rate to the austenitization temperature the austenitization temperature and time are the main factors determining the austenite grain size prior to quenching. The higher the austenitization temperature and the longer the austenitization time the larger is the average austenite grain size after austenitization. To obtain small average grain size usually the lowest austenitization temperature and time are selected. However, the selected austenitization temperature must be high enough to lead to a homogeneous solid solution of austenite as described above.

AUSTENITE CONDITIONING

Martensite forms by a displacive transformation from the parent austenite phase. Therefore, the grain and dislocation structure, or in general the defect structure is inherited to the product phase, because the relationship between neighboring atoms does not change. Thus the microstructure of the austenite is very crucial for the final properties of the martensite. It has been shown that austenite deformation prior to quenching can lead to an increase in strength without adverse effects on ductility or toughness. The deformation and the subsequent solid state reactions prior to quenching lead either to austenite grain refinement or to a subgrain dislocation structure. These
Fig. 5 Hot-working flow curve of austenite and mechanical properties after high-temperature thermomechanical treatment and tempering at 420°C for Fe-0.55C-1.6Si-0.6Mn (mass%) [25].

Microstructures have a positive effect on the final properties [25]. Therefore, it is important to understand how deformation within the austenite region controls the evolution of grain size and shape and generates a favorable dislocation substructure.

Fig. 2 presents hot-working flow curves. During hot working first the stress rises due to strengthening and later is compensated by softening. Depending on which mechanism is dominantly responsible for softening two different types of stress evolution with increasing strain are observed. If softening only takes place by dynamic recovery then curve 1 is observed. Flow curves of type 2 occur when softening at strains higher than the maximum stress is carried out by dynamic recrystallization. During dynamic recrystallization the softening leads to a drastic decrease of the dislocation density, whereas during dynamic recovery a stable dislocation rearrangement is the main mechanism of softening. Therefore, the stress decrease observed during dynamic recrystallization is larger. Fig. 3 shows the microstructure evolution for a material that is deformed in compression and recrystallizes dynamically. Shortly before the flow curve maximum the austenite grains are elongated and the primary grain boundaries are serrated [26]. The serration is correlated to the accelerated polygonization in the vicinity of grain boundaries. Around the maximum of the flow curve a small volume fraction of small recrystallized grains is observed at the primary austenitic grain boundaries. With increasing strain the volume fraction of recrystallized grains increases. Thus hot working offers a wide variety of microstructure control [Fig 4]. In case of thermomechanical treatment the aim is to optimize the mechanical properties. Therefore it is necessary to understand which austenitic structure results in optimized properties. It has been shown, that for low alloyed steel, deformation temperatures in the range of 900 to 950°C, strain rates of 3 to 20 1/s and strains between 25 and 40% the maximum of the flow curve is observed [25]. With increasing strength of the austenite not only the strength of the resultant quenched and tempered martensite increases but as well the toughness and the reduction of area.
Tempering at temperatures below Ac1 after quenching is performed to set the desired combination of mechanical properties. During tempering various solid state reactions take place. These are: stress relaxation, precipitation of carbides, decomposition of retained austenite, recovery of the martensite structure and grain boundary equilibrium segregation. With regard to the mechanical properties and their optimization the carbide precipitation has been the main focus. The embrittling effect of impurities has been minimized by limiting the bulk concentration of the impurity elements. The carbide precipitation is controlled mainly by the tempering temperature and secondly by the holding time at tempering temperature. Fig. 6 gives an overview over the possible reactions during tempering. The relationship between tempering temperature and mechanical properties is shown in Fig. 7. The ductility and toughness of quenched martensite decrease with increasing strength, i.e. with decreasing tempering temperature. Additions of impurity elements further reduce the ductility or toughness. Our work has shown that phosphorous, copper and tin additions significantly reduce the ductility over the entire range between 250 and 450°C. A drastic loss in ductility or toughness is observed in the lower temperature range where cementite starts to precipitate [1-5]. In this range cementite preferentially precipitates in form of thin films at martensite lath or grain boundaries. In case of impurity loaded boundaries, during loading the metal fails in an intergranular way.
Above the different austenite microstructures after deformation were presented. Because the austenite transforms without diffusion into martensite all the generated defect structures are inherited during quenching to the martensite lattice, they influence the carbide precipitation within the matrix and at the former austenite grain boundaries [27-29], and as well the segregation behavior. The result is a lower tendency for grain boundary segregation, a better distribution of impurity elements, refinement of the carbide within the matrix and at the grain boundary. These changes lead to a better ductility and toughness and allow the application of lower tempering temperatures, and hence higher strength can be achieved at the same level of ductility, toughness or endurance limit.

STRATEGY FOR OPTIMIZATION OF MECHANICAL PROPERTIES

After having discussed the basics principles of heat treatment and austenite deformation for high strength low alloy martensitic steels, a strategy for optimization of mechanical properties based on thermomechanical treatment will be presented. The necessary steps to be carried out during the optimization process are:

1) determination of CCT diagram with and without deformation
2) determination of optimum austenitization conditions with and without deformation
3) recrystallization behavior and post deformation solid state reactions
4) defining the optimum deformation temperature for optimum mechanical properties
5) final optimization including optimization of tempering temperature and combination of deformation steps

CCT diagrams for most commercial steels are available and well known. The important information to be extracted from such diagrams is the critical time needed for quenching from austenitization temperature to obtain a fully martensitic microstructure. However, conventional CCT diagrams may become less important, when the austenite is deformed prior to transformation. The respective
parameters of the thermomechanical treatment determine the state of austenite and hence influence the kinetics of the subsequent transformation and the microstructure of the product phase [30]. Deformation of austenite prior to quenching accelerates the transformation to ferrite and pearlite [30]. Homogeneously distributed dislocations can depress the martensite start temperature (MS), whereas localized shear bands will aid nucleation and raise the MS [30].

Optimum austenitization conditions have been theoretically and experimentally determined and proven in practice for steels used commercially. The dependency of grain size with austenitization temperature is well known and reflects itself in lower toughness and ductility properties with increasing austenitization temperature [21, 31]. For microstructures that are produced by deformation, the ductility or toughness can be independent of austenitization temperature (Fig. 8). If recrystallization of austenite is involved in the thermomechanical treatment the effect of austenite grain size produced during austenitization is minimized, because the resultant grain size is the consequence of the recrystallization during or after deformation and does not vary strongly with the austenitization temperature prior to deformation. Our investigations verified this for the steel Fe-0.55C-1.4Si-0.65Cr-0.65Mn. Streißlberger’s work [31] shows that the thermomechanical treatment applied leads to an improvement of strength and toughness properties. The thermomechanical treatment resulted in refined austenite grain size and a more homogeneous grain size distribution. As a consequence a refined martensite structure was observed after tempering.

Recrystallization and post deformation solid state reactions. The knowledge of recrystallization behavior is essential in defining the deformation temperatures for an optimum thermomechanical treatment. Through recrystallization austenite grain refinement can be achieved leading to improved toughness and ductility properties. Nowadays, recrystallization behavior can be investigated using dilatometers or similar devices and use theoretical approaches to define the critical strain for dynamic recrystallization for each deformation temperature [32]. The determination of the recrystallization behavior will accelerate the process definition for a thermomechanical treatment.
Any solid state reaction after deformation like recovery, recrystallization or grain growth process will affect the mechanical properties. Because the direct investigation of the austenite grain substructure for commercial alloys is almost impossible, mechanical testing for different conditions cannot be avoided. To retard any solid state reactions, which might negatively alter the mechanical properties microalloying elements such as niobium and vanadium can be used together with the thermomechanical treatment [7, 9, 33, 34].

The deformation temperature and strain together with the strain rate and time between deformation and quenching determine the austenite state prior to quenching. During process development for a specific steel one can investigate the effect of deformation on microstructure and mechanical properties separately. To understand the relationship between the developed microstructure and the mechanical properties, it is more convenient to combine the tests and relate the mechanical properties to the microstructure. Fig. 4 shows the variation of ultimate tensile strength and reduction of area for the steel Fe-0.55C-1.6Si-0.6Mn as a function of deformation strain. Streißlberger’s investigation confirms the dependency of strength on deformation strain (Fig.9). For 50CrV4 (Fe-0.54C-1.09Cr-0.97Mn-0.32Si-0.12V (mass%)) Peters demonstrated that both a recrystallized (deformation temperature TD=860°C) and a non-recrystallized (TD=770°C) austenite grain structure lead to a significant increase of the ductility while maintaining the same level of strength [33]. Samples were subjected to logarithmic strains in the range of 0.3 to 1.0. For tempering temperatures of 280, 380, 420 and 460°C the ultimate tensile strength was constant for all deformation strain.

Optimized thermomechanical treatment. In Fig. 10 different thermomechanical treatments are compared with the conventional heat treatment. As already described above, the thermomechanical treatment can result in a recrystallized or non-recrystallized austenitic grain structure prior to quenching. Compared to the conventional treatment, both austenite grain structures might result in
Fig. 11 Steel 55SiCr6 (Fe-0.55C-1.5Si-0.67Cr-0.59Mn with phosphorous between 0.0023 and 0.0213 and copper + tin (ratio 9:1) within 0.304 and 0.594 (all (mass%)). Comparison of mechanical properties of conventionally heat treated samples (CHT, black squares) and thermomechanically treated samples (TMT, blue circles).

superior properties. Then the question arises, which treatment should be selected. The selection of variables depends on the production limitations and on the design requirements of the part for which the steel is used. A work-hardened, non-recrystallized grain structure (route number 3) behaves more anisotropic than a recrystallized variant (route number 2) and might not be used in applications, where load directions other than the rolling directions might be important. This anisotropy is due to the prior austenite grain structures that are inherited to the final martensitic structures.

However, a sequence of deformations in some cases might be more beneficial for the properties. As shown in Fig. 10, the thermomechanical treatment might consist of two deformations. In a first step the austenite is recrystallized and a finer grain size is obtained. In a second step the recrystallized austenite is then work-hardened to increase the defect density prior to quenching. Martensite produced in such a way has two advantages. On the one hand the grain size refinement of the austenite results in a higher toughness and ductility and on the other hand the increased defect concentration that is inherited to the martensite, can lead to a finer dispersion of carbides during precipitation and to a higher solubility for impurities that are trapped by dislocations and vacancies. Such a material would then be insensitive (in a certain range) to impurity content variations (Fig. 11). For 55SiCr6 a two-step thermomechanical treatment combining recrystallization and subsequent work-hardenning exhibit superior mechanical properties with respect to conventionally quenched and tempered samples. Within the investigated concentration limits, the treatment applied is insensitive to the phosphorous or copper and tin content. As plotted in Fig. 11, for a specific tempering temperature the ductility of the conventionally heat treated samples strongly depends on the impurity content. Work at our institute shows, that for different spring steels a suitable thermomechanical can be defined that leads to both, improvement of strength and ductility. Additionally, properties such as the endurance limit or the toughness benefit from the
thermomechanical treatment [9, 33, 34]. More important the embrittling effect of elements such as phosphorous is minimized or eliminated.

CONCLUSION

Application of thermomechanical treatment where possible is a suitable method to improve the properties of high strength steels. The results presented here show clearly that deformation of austenite before quenching is beneficial for the mechanical properties. Thus, thermomechanical treatment opens up ways of improving existing steel grades without developing new alloys or implementing complex heat treatments.

Important factors for definition of a thermomechanical process are the deformation temperature, the deformation strain, the deformation speed.

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