Development of High-Strength Martensite by Age-Hardening of Coherent Intermetallic Precipitates

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Introduction

Only steels with a lath martensite microstructure offer a combination of high strength and good ductility. By a thermo mechanical treatment the mechanical properties can even be improved to a tensile strength of 920 MPa and a transition temperature of -160 °C /1/. These values could be achieved by a lath martensite containing 2 wt% Mn, 2 wt% Cr, 1 wt% Mo and 1 wt% Ni [fig. 1]. To garantie a complete lath martensite microstructure, the carbon content has to be limited to 0.03 wt%, which is too low to enable an evident increase in strength by precipitation of Nb- or Ti-Carbonitrates .

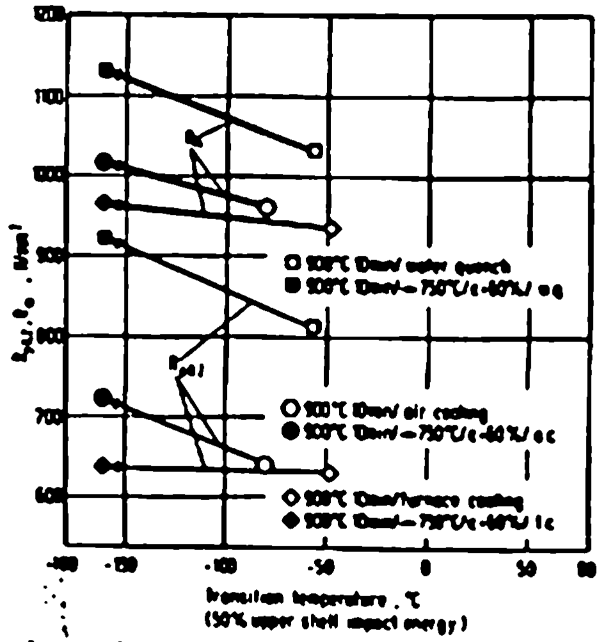


Fig. 1: Mechanical values improved by thermo mechanical treatment of lath martensite. 0.03 % C, 0.5 % Si, 2 % Mn, 2 % Cr, 1 % Ni, 1 % Mo after /1/

The only way to improve tensile strength in this steel, within an acceptable loss of toughness, is the age-hardening of lath martensite by intermetallic phases. Age-hardening by incoherent intermetallic particles, like in maraging steels, requires a high degree of supersaturation of alloying elements and as nucleation is connected with lattice defects, only inhomogeneous precipitation, mainly at grain boundaries, can be achieved. Contrary, coherent precipitates bring about a remarkable hardness increment even in low alloyed steels. Their homogeneous and fine dispersion garanties a good toughness. Ordered, coherent particles increase the flow stress even more than coherent particles, which can be sheared by single dislocations, as a high anti-phase boundary energy in ordered particles requires the formation of superdislocations. Furthermore, these phases have a low growth rate, stabilising their coherency.

In ferrite, it is possible to achieve a considerable hardness increment with small additions of Si and Ti by the precipitation of Fe_2SiTi. This phase has a $L2_1$ structure, being closely related to DO_3 Fe_3Si with twice the lattice parameter of the iron matrix /2/. As Si and Ti restrict the γ-Phase at high temperatures, Mn and Ni has to be added to achieve an entire α-γ transformation and, after cooling, a subsequent transformation into lath martensite. It has been possible to develope a variation of steels with relatively low additions of 2-4 wt% Si and Ti and 2-4 wt% Ni and Mn.

Mechanical Properties

Investigations to optimise the chemical analysis and the heat treating parameters have been carried out to ensure a) total transformation into γ, b) sufficient solubilitiy of the alloying elements to garantie maximum age-hardening and c) minimal grain growth. It was found, that specimens austenitised for 30 min at 1100 °C offer the highest hardness increment and a fine grained microstructure, so that both, good ductility and high strength could be expected.

An adequate age-hardening is possible in the temperature interval between 400 °C and 700 °C. 500 °C is the most suitable aging-temperature for most of the alloys, achieving a satisfactory maximum hardness of 560 HV within an acceptable annealing time of 2 hours. The hardness decreases only after more than 22 hours of annealing, while the hardness of a non-hardenable lath martensite did not have any alteration in hardness during annealing below 600 °C [fig. 2].

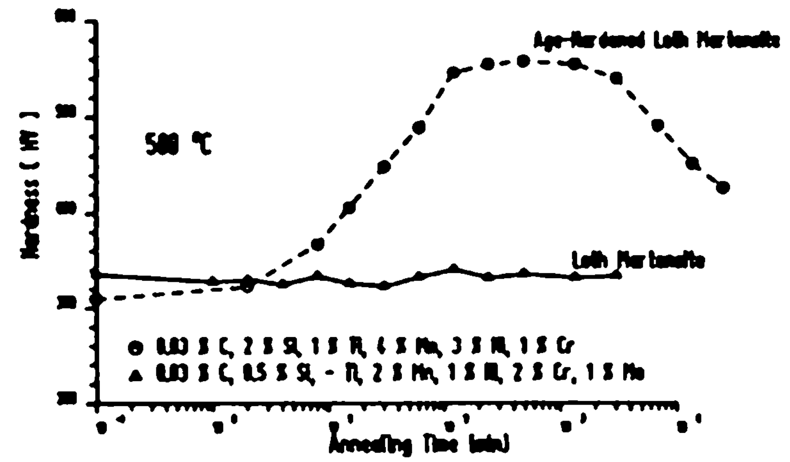


Fig. 2: Variation of hardness with aging time of age-hardened lath martensite

Compression tests showed, that with increasing annealing time the tensile strength and hardness rise to a maximum of 1800 MPa, i.e. 560 HV, while the total elongation, as an indication of ductility, decreases from 65 % to 15 %. In the over-aged condition, as the elongation is rising, the tensile strength and hardness decrease rapidly, while the ultimate strength remains at a high level [fig. 3]. The tensile strength can be raised to almost the value of maraging steels, while the toughness of the high alloyed maraging steels can not quite be reached. Specimens at peak-hardness and underaged specimens showed after compression distinct slip steps so that a coarse distribution of slip bands can be expected as reason for the low values of ductility.

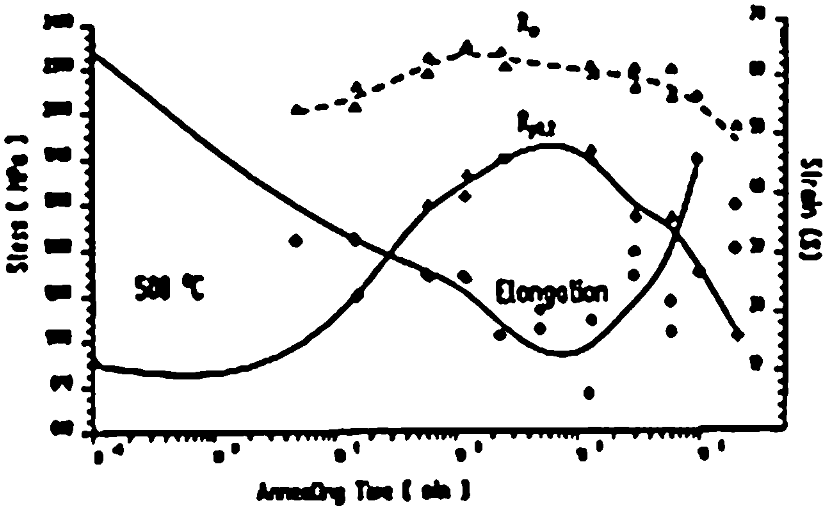


Fig. 3: Variation of mechanical values with ageing time, measured in compression test.

Fractography of aged specimens after compression showed a mainly brittel fractured surface. Only specimens in the overaged state had small parts with traces of plastic flow. The brittel fracturs were mainly intercristalline but also transcristalline, whereby the intercristalline cracks could not be related to particles or phases on grain boundaries.

Thermo Mechanical Treatment (TMT)

The thermo mechanical treatment, which was developed for these steels, makes it possible to transform the deformed austenite without prior recristalisation into a highly destorted lath martensite [fig. 4]. To ensure sufficient solid solution of the alloying elements at still acceptable austenite grain size, the austenitisation was limited to 30 minutes at 1100 °C. A hot deformation rate of $\phi = 1$ and even $\phi = 1.5$ is possible without cracking, if the precipitation process, which causes embrittelment, was avoided after TMT by instantaneous water-quenching. The deformation temperature could be reduced to 700 °C.

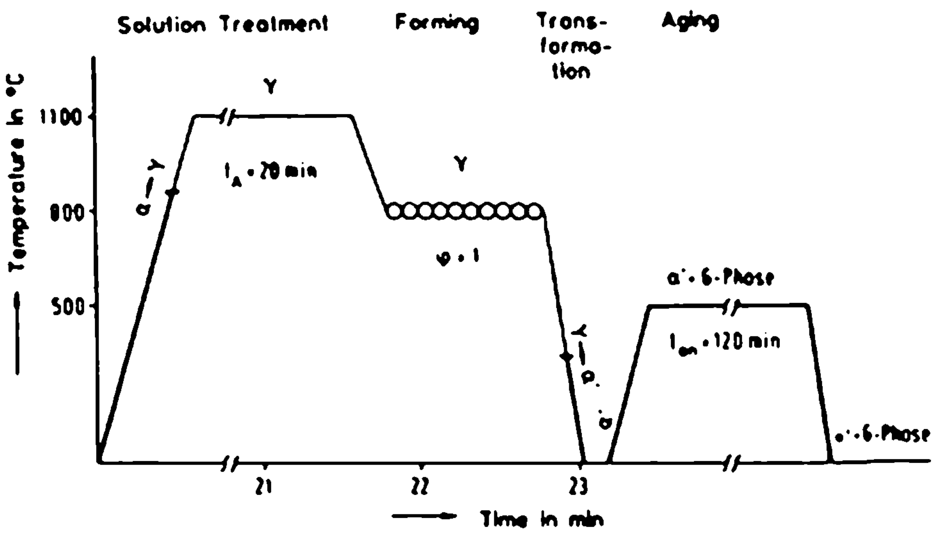


Fig. 4: Schematic presentation of the specially developed TMT.

Fig. 5a: Microstructure without TMT.

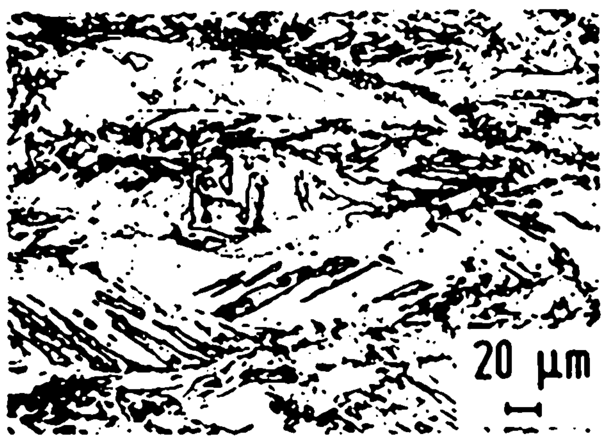


Fig. 5b: Microstructure after TMT.

Fig. 6a: Electron micrograph of lath martensite micro-structure without TMT.

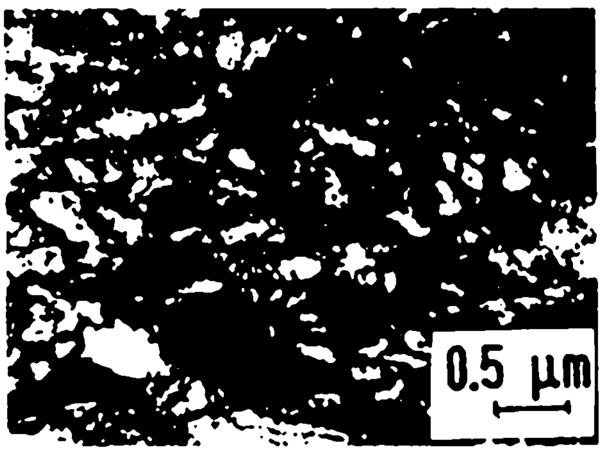


Fig. 6b: Electron micrograph of lath martensite micro-structure after TMT.

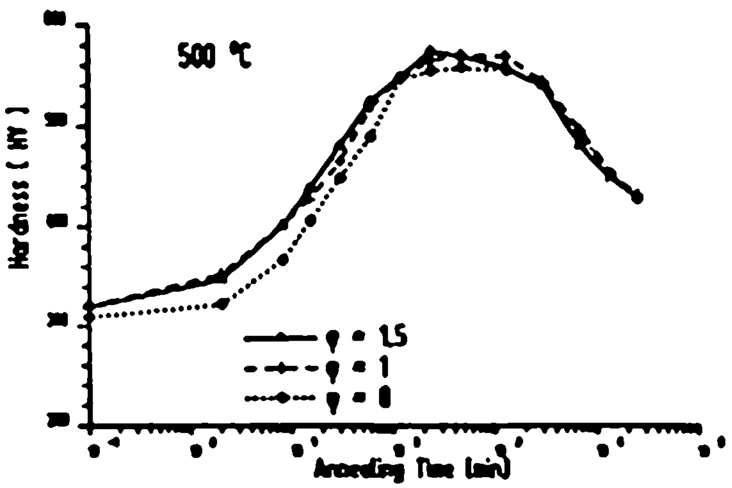
The microstructure of thermo mechanical treated specimens show flatt, pancake-shaped prior austenite grains, finer packet size [fig. 5 a-b], shorter laths and a higher dislocation density than those without thermo mechanical treatment [fig. 6a-b]. The influence of the TMT on the hardness is relatively small. The hardness increases faster and the maximum hardness is higher than without TMT [fig. 7]. In compression tests the same effect on the 0.2 % proof stress was measured, while the ultimate tensile strength is hardly influenced [fig. 8]. The elongation, as a criterion for ductility, is little improved by TMT, but the measurements show a significantly lower standard deviation after TMT [fig. 9]. To conclude, the increase in ductility by TMT measured for the basic lath martensite, which is due to a homogeneous and fine lath structure and high, homogeneous dislocation density, could not be achieved in age-hardened lath martensite by an even more optimised thermo mechanical treatment. It can be supposed that the influence of the precipitation hardening dominates the influence of fining the lath structure.

Examination of the precipitates

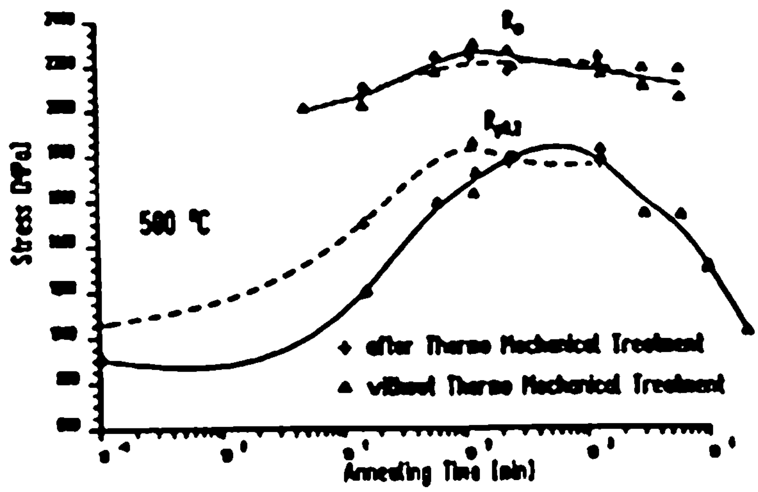
In electron microscopical investigations of the new Ni and Mn containing martensitic alloys the cuboidal, fcc G-Phase $(Fe,Ni,Mn)_{16}Ti_8Si_6$ /3/ [fig. 10] and not the spheroidal Fe_2SiTi /2/ [fig. 11] was found. The G-Phase has a lattice parameter four times of that of ferrite, is also coherent to the matrix and seems to be a complex version of the DO_3 structure, composed of 8 DO_3 unit cells /4/.

The precipitates are finely dispersed and homogeneously distributed in the matrix. As they are coherent, their orientation is dependent on the matrix orientation [fig. 12]. The precipitates do not seem to be nucleated at dislocations, although, in grain boundaries larger precipitates are found [fig. 13]. They become coarser with increasing annealing time. Denuded zones can not be observed. This arrangement of particles can be explained by homogeneous nucleation. The different size may be caused by faster diffusion in the grain boundary than in bulk material.

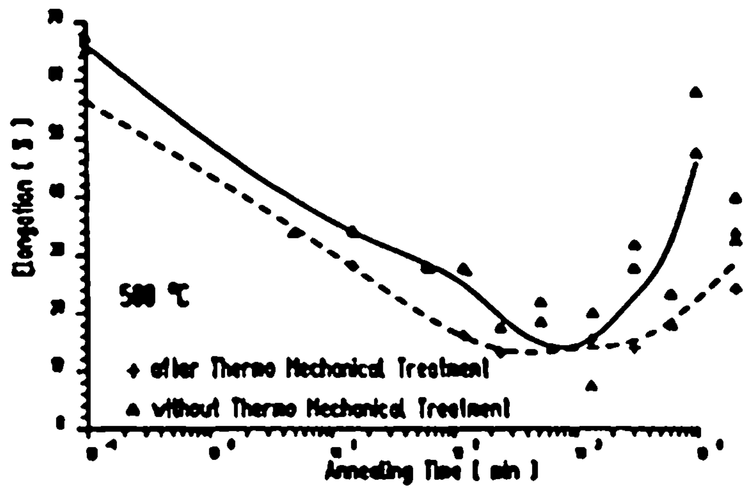
To investigate the influence of the particle size on the hardness, specimens at characteristic stages of the ageing process at 500 °C are analysed. At the maximum hardness after 2 hours annealing at 500 °C, the average particle diameter is less than 5 nm, while after an annealing time of 22 hours, at the begining of the hardness curve's decline, the diameter is only slightly more than 5 nm. In



<u>Fig. 7</u>: Variation of hardness with ageing time and deformation rate.



<u>Fig. 8</u>: Influence of THT on strength in compression test with ageing time.



<u>Fig. 9</u>: Influence of THT on elongation in compression test with ageing time

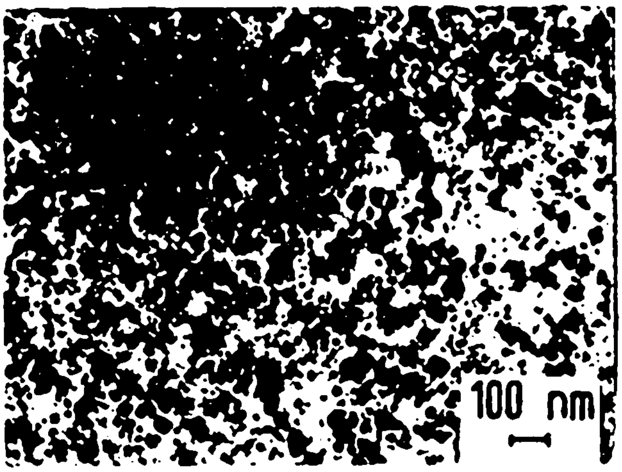


Fig. 10 : Extraction replica, G-Phase $(Fe,Ni)_{16}Ti_8Si_6$

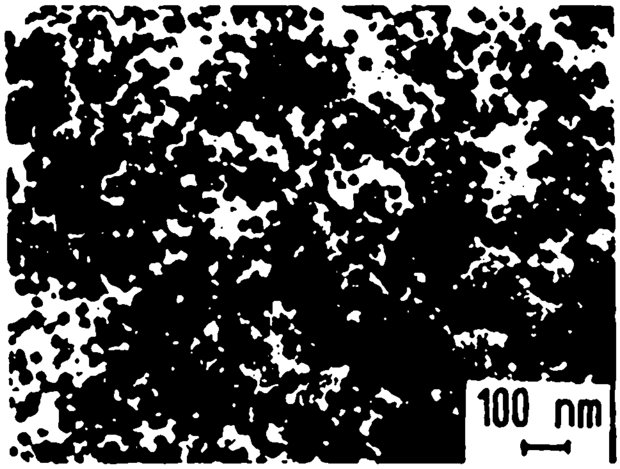


Fig. 11 : Extraction replica, Fe_2SiTi spheroidal morphology

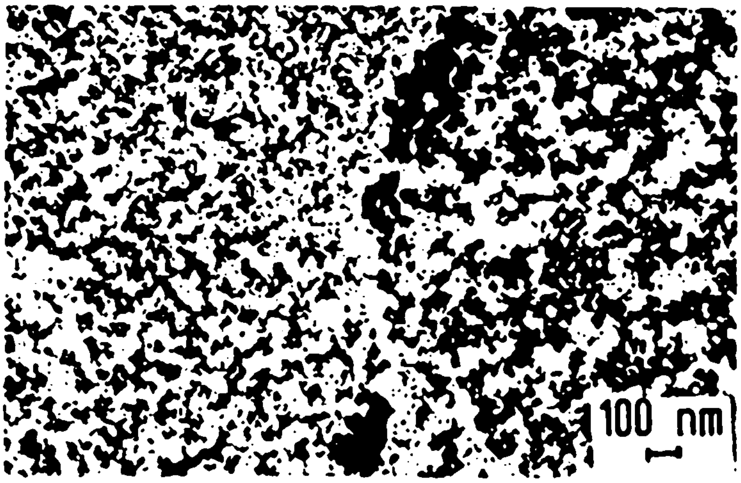


Fig. 12: G-Phase particles of two diffrently orienated grains, extr. replica

Fig. 13: Extraction replica, showing coarse precipitates on grain- and packet boundaries

the overaged condition after 95 hours the average particle's size is still only 8 nm but their shape is more expliced cuboidal. Even in the completely overaged state the precipitates are perfectly coherent to the matrix and no traces of strain contrast were found. At peak hardness the precipitates show, even in dark field image, no distinct phase boundaries, while their diffraction patterns can be clearly distinguished. One reason can be, that in this state of the precipitation process there are ordered zones or phases with the composition Ni_2TiSi as were postulated by Utsunomiya /5/ or nucleii of Fe_3Si incorporating Ti atoms as supposed by Brown /6/ in Fe-Si-Ti alloys.

Summary

Precipitation of ordered, coherent, finly dispersed precipitates produce a signifficant age-hardenig. This is connected with a loss of ductility, caused by inhomogeneous slip, which gives slip bands, formed by previous shearing of the coherent particles. In return, overaged specimens have an acceptable proof strength of 1400 MPa as well as suitable ductility. In this state the precipitates are still very small with a diameter less than 10 nm and the plastic flow may be controled by Orowan bowing of dislocations between precipitates.

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