Budapest University of Technology and Economics
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THE EFFECT OF MICROSTRUCTURE AND DEFORMATION OF ALUMINIUM- KILLED LOW CARBON ENAMEL-GRADE STEEL SHEETS TO THE INTERACTION BETWEEN STEEL AND HYDROGEN

Summary of the PhD theses

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This dissertation, the comments of the reviewers and the log recorded on the defence are on show in the Decan's office of the Faculty of Mechanical Engineering of the Budapest University of Technology and Economics

1. **Introduction**

It is well known that the fish-scale formation on the surface of enamel coated steel products is caused by appearance and recombination of hydrogen at the sheet-enamel interface. The hydrogen can be absorbed in the metal during the enamel firing. The tendency for fish-scale formation in case of the cold rolled Al-killed low carbon enamel grade steel sheets is characterized by the hydrogen permeability. That is why hydrogen permeation test has been used for long time to estimate the hydrogen absorption capability of steel sheets used for enamelling. In order to avoid the appearance of fish-scale on the enamelled products for conventional cold rolled, Al-killed low carbon enamel-grad steel the hydrogen permeation time has to be $T_H = \frac{t_0}{d^2} \geq 6.7$ where „t_0" is the hydrogen permeation time [min], "*d*" is the thickness of the steel sheet [mm].

The hydrogen is not homogeneously distributed in steel, as it would be in perfect iron crystal. In steel various trapping sites exist for hydrogen atoms. Hydrogen will be found not only in the normal interstitial lattice sites but also in atomic and micro-structural imperfections such as vacancies, solute atoms, dislocations, grain boundaries, boundaries of second phase particles, steel-inclusion interfaces and microcavities. The general term for this phenomenon is trapping. Trapping enhances the solubility of hydrogen but decreases the diffusivity (*daSilva, 1976, Pressouyre,1979, Oriani, 1978, Hirth, 1980, Pressouyre and Bernstein,1981 Gibala 1984, Kiuchi és McLellan 1986,* McMahon, 1990*, San-Martin ,1993; Grabke, 2000*).

Traps can be characterized by their nature. Some traps do not change during of technological operation (i.e.: solute atoms, boundaries of inclusions during annealing), other traps is capable to change considerably (i.e.: grain boundaries or precipitations). However, because of its complexity, it remains an unsolved problem.

The microstructure of steels differs from the high purity iron, so there are other tapping sites too for the hydrogen. At Al-killed low carbon enamel-grade steel sheets, in order to avoid the fish-scale on the enamelled products, the microstructure of the hot rolled strip should be formed from ferrite and carbides (*Verő, 1994*). At these steels the microstructure is formed from ferrite and large carbides when the coiling temperature of the hot rolled sheet is high.

In the pure iron the ferrite grain boundaries trapping effect is not clear. After Lee and Lee (*Lee and Lee, 1986*), Ono and Meshii (*Ono and Meshii, 1991*) the grain boundaris are strong trapping sites. After Hagi and co-workers (*Hagi, 1979*), Choo and Young (*Choo and Young, 1982*) are week trapping sites. After Kiuchi and McLellan (*Kiuchi., 1986*) the trapping effect of ferrite grains boundaries are negligible. After Gesari and co-workers (*Gesari, 2002*) the ferrite grain boundaries have medium trapping effects on hydrogen. Martinez Madrid and co-workers observed that the ability of the grain boundaries in iron to trap hydrogen is a function of the grain boundary angle. A coarse grain microstructure, with high angle grain boundaries occludes large amounts of hydrogen and has the highest susceptibility to hydrogen embrittlement. Matsumoto and co-workers found a good correlation among, grain boundary energy, grain boundary free volume, and the hydrogen concentration at grain boundaries under hydrogen environments: high-energy grain boundaries have large gaps, and many hydrogen atoms are captured at these spaces. Thus, higher-energy grain boundaries are more influenced by hydrogen. The binding energy between hydrogen and the grain boundary is negligible under the assumption that carbon and nitrogen atoms are fully segregated at grain boundaries at their solubility limit. Therefore, carbon and nitrogen atoms exclude hydrogen atoms from the grain boundaries, and improve the cohesive energy of the grain boundaries under hydrogen environments

For a long time there has been controversy over the extent of the effect of cold working, as discussed by Marandet (*Marandet, 1977*). There was reported that at unalloyed carbon steels plastic deformation usually increased the total amount of hydrogen occluded, but indicate a disagreement in the literature over the associated hydrogen embrittlement phenomenon, many investigator finding a deleterious effect, others a beneficial effect. After Szklarska-Smialowska and Z. Xia *(Szklarska,1997*) the presence of dislocations is responsible for the hydrogen diffusivity in iron at room temperature. H. Huang et al. (*Huang,1995*) examined hot rolled type 1020 steel (C=0.18-0.23, Mn=0.3-0.6). At this steel H diffusivity decreased with increasing cold work and levelled out when cold work reached 30% to 40%. Keth [14] observed that the dislocation density increased with cold work and levelled out when cold work reached 30% to 40%. Kumnick and Johnson *(Kumnick and Johnson,1980)* studied high purity iron, deformed by cold rolling at room temperature. After they the trap density in annealed iron was $8.5x10^{20}/m^3$, after 15% cold deformation was $5.9x10^{22}/m^3$, after 30% was $5 x10^{22}/m^3$, after 40% was $7 x10^{22}/m^3$ after 60% was $1.5x10^{23}/m^3$. They suggested that dislocations in pure iron were the source of the H trapping behaviour. P Alexandru(Alexandru, 2004, Alexandru ,2005) observed at cold rolled rimmed steel for deep drawing and enamelling type A3 (*STAS 9485-80*) (C $\leq$ 0.08, Mn=0.2-0.45, Si=0.03-0.08) that after ε=20% reduction of thickness appeared fish-scales, and maximum number of fish-scale/m^2 appeared at 30% deformation degree. Similarity were observed in hydrogen permeability before (*Albert, 1975*).
A Juan (*Juan, 2000; Juan, 2001*) considered that H accumulation could be less favourable near a screw dislocation than an edge dislocation Martinez-Madrid et al.(*Martinez-Madrid, 1985*) observed that in general the hydrogen content increased with the degree of cold work at cold worked iron, but there was a drop in the hydrogen content of specimens with 5-10% of cold work. They observed very little scatter in the result for hydrogen content for small amount of deformation, and as deformation increases there were greater variation in the hydrogen content. Simonetti et al. (*Simonetti, 2003*) observed the hydrogen does not accumulate in the neighboring of the carbon. The carbon acts such as expeller of hydrogen. The a/2[$1\bar{1}1$] dislocation creates an energetically favorable zone for accumulation of carbon. The presences of carbon in the dislocation core make no favourable hydrogen accumulation**.**

Research objectives

1. In literature were observed, that, the TH values are different at different positions of the coils at Al-killed, low carbon, unalloyed enamel grade steel sheets. By EN10209:1996 standard the edge of the cold rolled, Al-killed, unalloyed, low carbon enamelling grade steel sheets are the most succeptibile for fish scale forming, but the reason were not revealed yet. What kind of traps are responsable for the long hidrogen permeation time at DC01EK and at DC04EK steels?

2. The technological operations have complex effect on the traps, which coexist in the enamel grade steels. Ther were few data about the cold rolling effect on microstucture and on T_H value at hot rolled unalloyed low carbon Al-killed steel sheets. Appeared the question how change the hydrogen permeability and the microstructure of EK2 and EK4 steels during the cold rolling.

3. The hydrogen can be absorbed in the steel during the enamel firing. There were no study before of the effect of the enamel firing thermal cycle on the microstructure and on the hydrogen permeability of the steel sheets.

4. The enamelled products more or less are cold deformed before enamelling. In the literature are lot of contradiction about cold rolling effect on fish scale succesibility. There were the objective to study the cold deformation effects on T_H value and on the microstructure of cold

rolled, Al-killed, unalloyed, low carbon enamelling grade steel sheets with different T_H values in delivery conditions.

3. Materials and testing

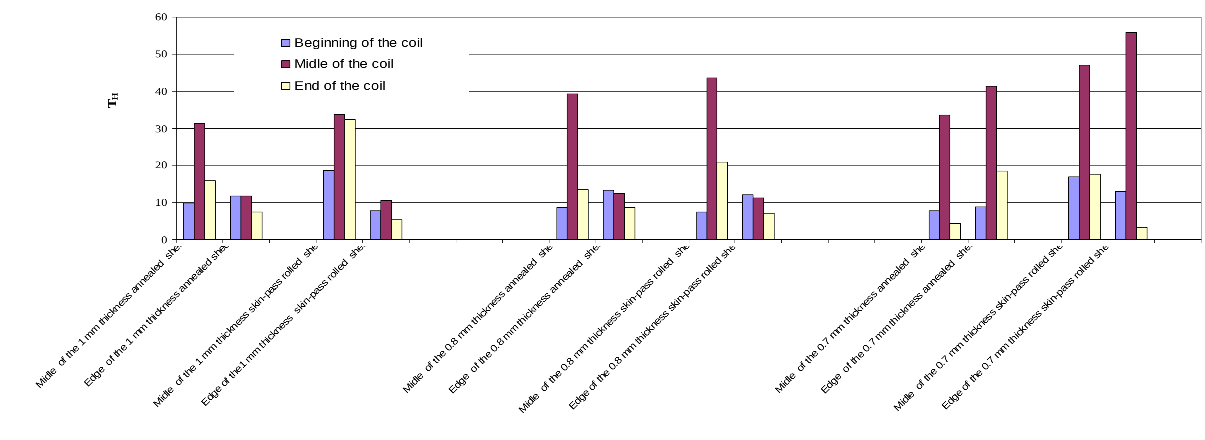
My theses concern to low carbon, unalloyed, Al-killed cold rolled steel sheets type DC01EK and DC04EK (by EN10209:1996 standard), and on the base material of these sheets: high temperature coiled EK2 and EK4 hot rolled quality sheets. The hydrogen permeability and microstructural properties were studied on same positions of the hot and cold rolled sheets.
To study of the effect of the microstructure of Al-killed low carbon enamel-grade steel sheets on hydrogen permeability different zones of three cold rolled, Al-killed low carbon enamel-grade sheet steels were examined after annealing and after skin-pass rolling. Each of the examined steel sheets was elaborated in LD steel converter, followed by continuous casting, hot rolling and pickling. The coiling temperature of EK2 an EK4 strips were 730 ±15°.Tthe hot rolled sheets were cold rolled in industrial condition by quarto mill stands at Dunaferr-Voest Alpine Ltd. The examined coils were annealed (670°C/ 16 hours) in gutter-wound coil, in bell-type annealing furnace. The annealing was succeeded by skin-pass rolling as the steel would be sold to enamelling companies..
For detailed study of cold rolling effect on the hot rolled, non alloyed, Al-killed low carbon strips several samples were cold rolled from: EK2 quality strip in laboratory condition by duo rolling mill.
To study of effects of enamel firing, from each studied DC01EK and DC04EK quality sheet were cut out three specimens each of them in size of 70x90 mm. After degreasing they were joined by laser welding. The specimen received in this manner was coated with test enamel slurry and fired at 830^0C, 5 minutes in industrial conditions. After firing the specimens were cut by laser. The testing procedures were evaluated on the middle plat from the sandwich specimens.
The products are cold deformed with different *manner* at enamelling companies before enamelling. To study the cold deformations effects on the hydrogen permeability and on the microstructures samples were cold rolled on several rolling levels by duo rolling mill and quatro rolling mill in laboratory.
The hydrogen permeation time was determined by DIPERMET-H hydrogen permeability measuring equipment (developed by Bay Zoltán Institute for Material Science and Technology together with FQZ Brandenburg) In the DIPERMET-H device two carefully degreased specimen (40x70 mm^2) can be placed in a measuring cell with sulphuric acid electrolyte above them. In 6% H_2SO_4 aqueous solution were added 0.5 milligrams/litre As_2O_3 and 0.3 milligrams/litre $HgSO_4$. As_2O_3 added to the sulphuric acid solution prevents the recombination of hydrogen on the surface, while the Hg layer formed on the surface of the sheet increases hydrogen activity. The used count electrode was Pt. A semiconductor-based hydrogen detector located on the other side of the samples detects the over passing hydrogen.
The optical microscopically examinations were carried out on longitudinal, transversal cross sections and parallel to the surface of the sheet by a LEICA MF-4 microscope. The carbides morphologies were determined applying Quantimet 550 image analyser.
Electron backscatter diffraction (EBSD) was used to determine the individual grain orientations low angels, high angles grain boundary and characteristic fibres for the BCC rolling structure. An EDAX-TSL orientation imaging microscopy (OIM) system was used which was mounted on a Philips XL-30 scanning electron microscope. The acceleration voltage was 25 kV. Orientation image maps were constructed from about 30 000 pixels.
The transmission electron microscopically examination werw carried aut by an JEOL JEM 200A electron microscope.

The dislocation densities were determined by X-ray measurements which were carried out in a special high resolution double crystal diffractometer, type Philips PW 1830, operated at a rotating cobalt anode with fine focus. The samples were studied parallel with the rolling plane prepared for microscopically and for EBSD study after a new electro polishing.

Short summary of results

Effect of the microstructure of Al-killed low carbon enamel-grade steel sheets on hydrogen permeability

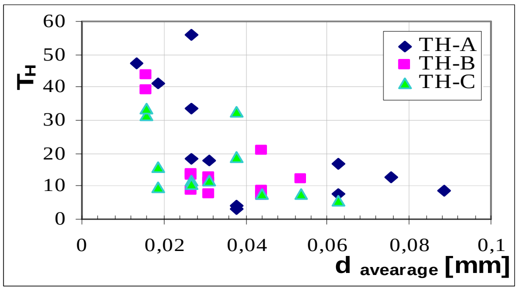
The T_H values of the samples taken from the middle of cold rolled coils, and from the middle of the transversal strips were long; but generally the T_H values of samples taken from the end or from the beginning of the coils, were much lower.



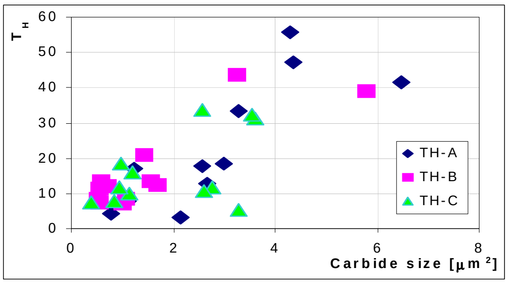
*The T_H value at different positions of the coils***.**

The microstructures of the samples were formed from ferrite, carbides and non-metallic inclusions. The microstructures of the cold rolled sheets were not homogeneous.

At the samples where big massive carbides could be observed (average areas higher than 4 μm^2), the hydrogen permeation times were long. The small, rounded carbides (with average area smaller than 2 μm^2) have no effect on hydrogen permeability even if they are present in very big number. When the average carbide size was about 2-4 μm^2 the T_H values depend on the number and morphology of the carbides, and/or if there were microcavities in the microstructure. When the carbides sizes were on average about 2-4 μm^2 and the amount of carbides were significant the T_H value were high. In case of reduced number of carbides T_H values were smaller (*Fábián, 2008*). Moreover microcavities between the carbides could be observed by optical microscope if the hydrogen permeation time was long.



Relationships between the T_H values and grain sizes

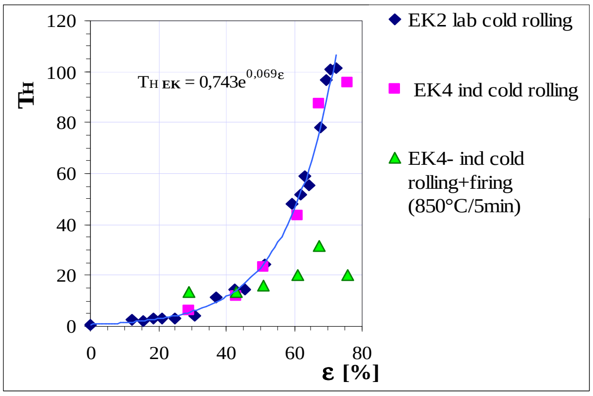


Relationships between the T_H values and carbide sizes

The ferrite grains size has no significant effect on T_H value. Although where the T_H values were higher, the ferrite grain size were fine, in each case there were massive carbides groups, and between the carbides could be detected micro-cavities. Where the ferrite structures were coarse-grained, there were lots of small discrete carbides, which did not form groups, and between the carbides there were no micro-cavities, the hydrogen permeation time was long.

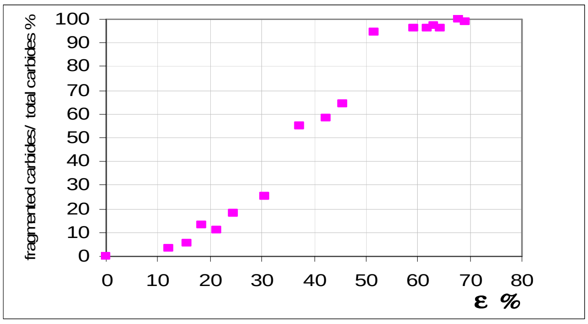
Cold rolling effects on EK2 hot rolled steel sheets microstructures and on the T_H values

Studying the cold rolling effect on the hydrogen permeability of the EK2 type hot rolled sheets were observed the T_H values increase exponentially by the thickness reduction as a result of the parallel microstructural changes during the cold rolling process. The microstructure of the hot rolled EK2 sheet was formed from ferrite(98±1%) and large massive carbides (2±1%). The non ferrous inclusions were 0 and 1 grade of oxides.

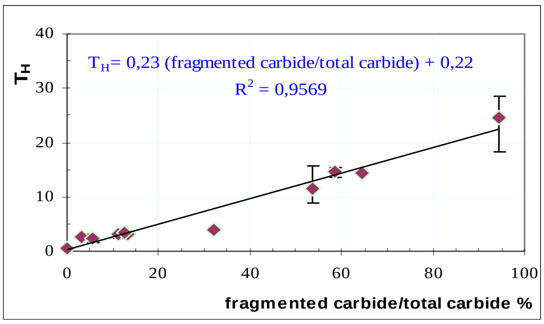


The cold rolling effect on the T_H value

Up to 31% reduction of thickness no significant changing in the microstructure was observed, but already after ε=12% it could be discern a few cracks in some of the carbides Studying by orientation image maps was observed that hot rolled steel is untextured. Still 31% reduction of thickness were not observed significant changing in the microstructure by microscope, but the pole density of <111>║ND texture increase still this level of deformation and the T_H value increase with the thickness reduction also. After ε=37% were observed numerous fragmented carbides and microcavities between them. The intensity of <111>║ND texture decrease comparing with the texture of specimen after ε=31%. At ε=51% each coarse carbide has became fragmented. As the fragmentation of the carbides increase in number the randomly oriented measured points quantity increase.

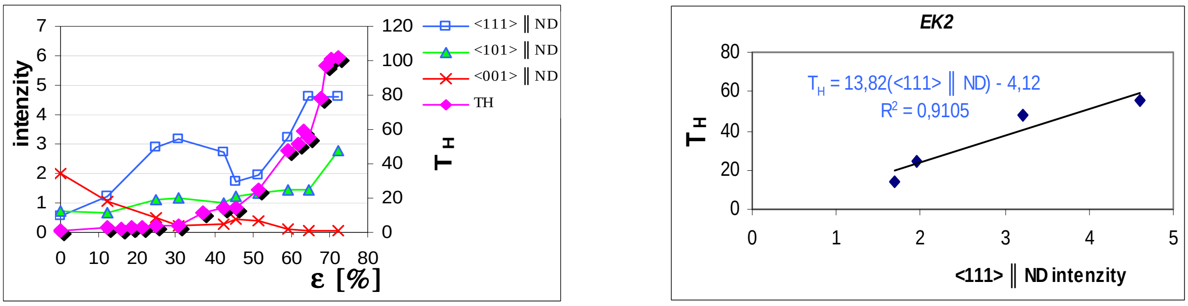


The ratio of (fragmented carbides)/(total carbides) as the function of deformation



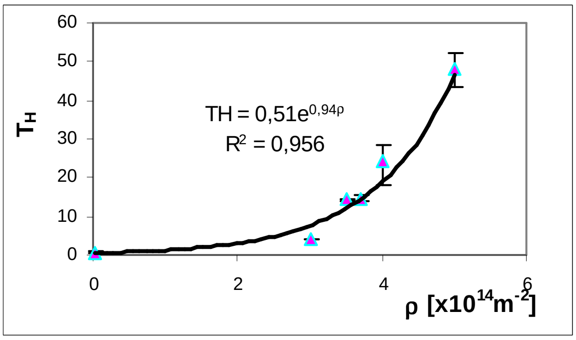
The ratio of (fragmented carbides)/(total carbides) effect on T_H value

When each of the carbide was fragmented increasing the deformation level increase as the intensity of <111>║ND texture such the hydrogen permeation time. There the T_H value increse linear with <111>║ND texture intensity



The effect of the cold rolling on texture and hydrogen permeability at EK2 strip

During the cold rolling the dislocation density increased from 10^{12} m^{-2} magnitudes to more than two magnitude orders *(Fábián, 2010a)*, The T_H value stil ε <60% increase exponential by dislocation density

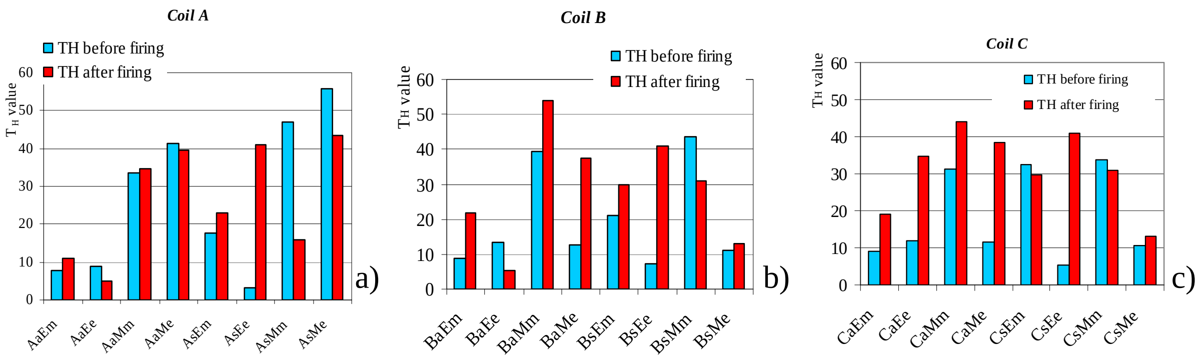


The dislocation density effect on T_H value at EK2 hot rolled steel

To distinguish the dislocation density trapping effect from the other trapping sites were measured the hydrogen permeability of specimens cold rolled in industrial condition and treated in enamel firing condition. The T_H value of heat treated samples, after high deformation degree, decrease to T_H value of cold rolled samples with ε =30% cold rolling degree

Effect of a firing thermal cycle on the microstructure and on the hydrogen permeability of Al-killed low carbon, unalloyed, enamel-grade steel sheets

The hydrogen can be absorbed in the metal during the enamel firing. There were studied the samples studied before after annealling (*notification: a*) and aftere skin-pass rolled (*notification: s*) in the middle of the coils (*samples notification: M*) and from the end (*samples notification: E)* as in the middle (*samples notification: m*) such at the edges (*samples notification: e*) of the transversal bands of sheets. Generally after enamel firing thermal cycle the normalised hydrogen permeation time were higher than before firing. After fireing each of the skin-pass rolled sampels T_H value was higher than 6.7 minutes



Effect of enamel fireing thermal cicles on T_H values
a)Coil A with 0.7 mm thickness b)Coil B with 0.8 mm thickness
c) Coil C with 1 mm thickness

During enamel firing thermal cycle the microstructure of the samples changed. After firing we could not develop big groups of massive carbide in microstructure. The microstructures

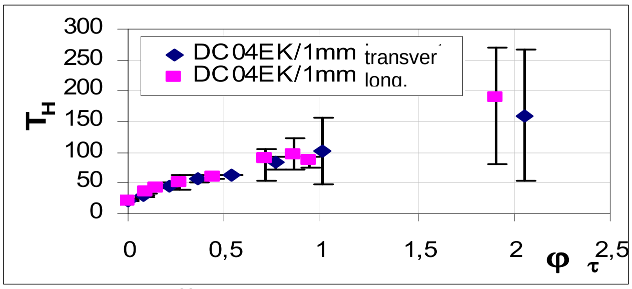
consist of ferrite, fine lamelar pearlite, sometime micro-cavity, cementite films on the grain boundaries, a few not transformed massive carbides and non-metallic inclusions. After firing the ferrite grain sizes of the samples became more uniforme.
By transmission electron-microscopical study were obseved fine (diameters are smaller than ~100 nm) disspersed precipitates inside of ferrite grains. The pearlite colonies at ferrite grain presented fine lamellar structure (the cementite and the ferrite layers thickness were ~20-60 nm).

Cold deformation effects of DC04EK steel sheets

There are lot of contradictions about cold rolling effect at Al killed , low carbon enamel-grade steels on hydrogen permeability, fish scale succesibility.
For comparability of various kinds of cold deformation were used the comparative deformations, which were calculated by:

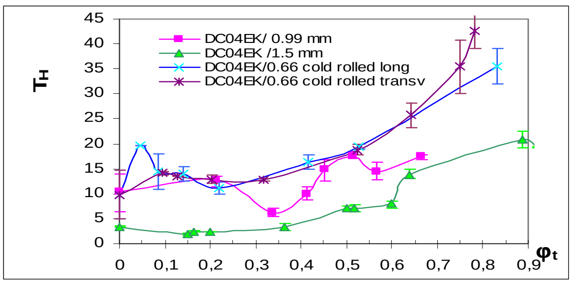
$$\varphi_t = \frac{\sqrt{2}}{3}\sqrt{(\varphi_x - \varphi_y)^2 + (\varphi_y - \varphi_z)^2 + (\varphi_z - \varphi_x)^2}$$

At the specimens DC04EK/1mm, where in delivered state the average T_H value was about 20 minutes, the fragmented carbides were situated at ferrite grains boundary, the T_H value increased with the degree of cold work. There was very little scatter in the result for T_H value for φ_t=0– 0.7. At samples, with φ_t>0.7 the T_H value in each case was higher than 50 min.

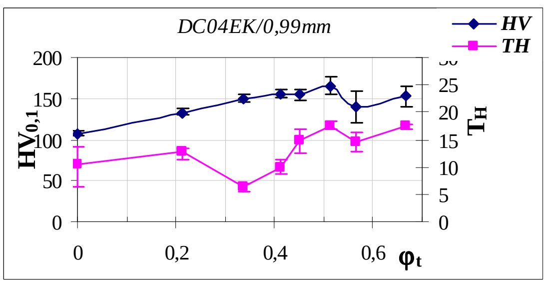


Cold rolling effect on T_H values at DC04EK/1mm sheet with $T_{H\varphi=0}$ ~20min

During of the cold rolling ofDC04EK/1.5 mm specimens, with low T_H value in delivered condition, where the carbide were small and round with size under 2 μm², and were situated inside of the ferrite grains, after slightly deformation level the trapped hydrogen quantities decrease similar to the pure iron (*Martinez-Madrid, 1985).*
In case of sheets with average T_H value ~ 10 minutes in delivered state the cold deformation effects on T_H value were absolutely different. During of the cold rolling there appear local maximums and local minimums in T_H value. The most interesting case appeared at DC04EK/0.99 mm sheet, where the average carbide size was 2-4 μm², and the carbides were situated inside of ferrite grains. At DC04EK/0.99 mm sheet after φ_t=0.21 comparative deformation the average T_H value was 12.7 min/mm², after φ_t=0.34 the T_H value decreased to 6.2 min/mm².) There were no scattering. Measuring the microhardness it was observed that at these samples increasing the deformation degree increase the hardness.

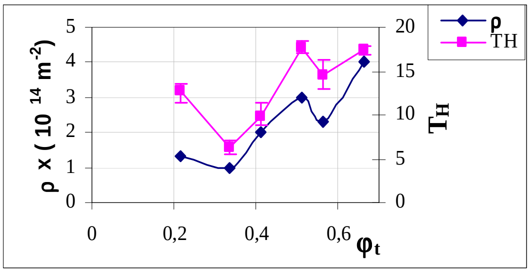


The cold rolling effect on T_H value at DC04EK samplesvith low and medium T_H value in delivered condition

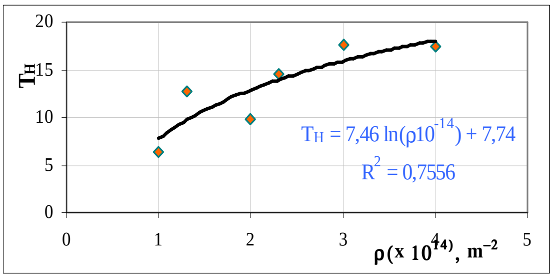


The cold rolling effect on T_H value and on the microhardness at DC04EK/099

Studying the dislocation densities determined by X-ray measurements and the T_H value of the samples there were observed that the T_H value increase or decrease with dislocation densities. At the local minim in T_H value (φ_t=0.34, ε_z=25%) the dislocations character was screw; at each other samples the dislocation character was mixed.

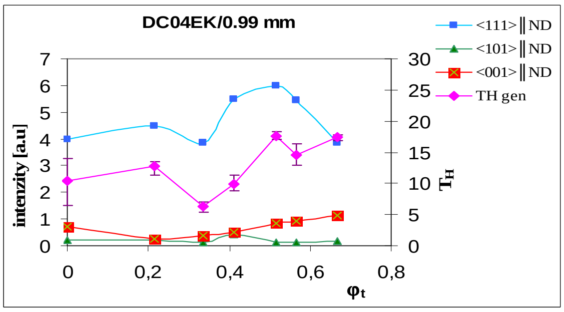


Cold rolling effect on the dislocation density of DC04EK/099 sheet and on T_H value

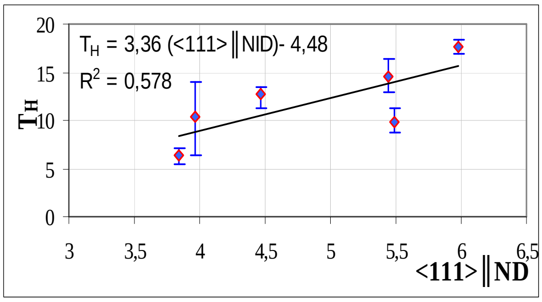


The dislocation density [m^{-2}] effect on T_H value at DC04EK/099 sheet

Studying the cold rolling effect on texture parallel with the rolling plane at samples DC04EK/0.99 mm at the beginning of the deformations (φ_t=0–0.51) the <111>‖ND texture intensity increase and decrease in similar way as the T_H value



Cold rolling effect on the texture of DC04EK/099 sheet and on T_H value



The <111>‖NID pole denity effect on the of on T_H value DC04EK/099 sheet

Theses

Thesis 1st

I have concluded that between the permeation time of hydrogen (T_H) and the microstructure in case of DC04EK and DC01EK type steel sheets:

- if the average area of carbides is bigger than 4 μm^2 in the polished plane, the T_H value is longer than 35 minutes, which clearly satisfies the conditions of scale-free enamel ability, where T_H is ≥ 6.7 minutes as for the MSZ EN 10209: 2000.
- if in the polished plane the average area of the fractured carbides is between 2-4 μm^2, the normalized permeation time of hydrogen changed between 3.3 to 43.5 minutes. The T_H value is longer if there are microcavities between the carbides.
- if in the polished plane the average area of the carbides is smaller than 2μm^2, the TH value is shorter than 25 minutes. In this case, the T_H value is irrespectively of the size of the carbide/ferrite interface per unit surface area.
- in case of those samples, of which the ferrite-typical grain size degree is smaller than G6 (d_m> 44.2 mm), moreover, small, rounded carbides can be found inside the ferrite, the T_H value is shorter than 25 minutes.

(Fábián, 2004a; Fábián, 2005; Fábián, 2006a; Fábián, 2008a; Fábián, 2008b)

Thesis 2nd

I have concluded that during the cold rolling process of the EK2 type hot rolled sheets, as a result of the parallel microstructural changes the T_H value exponentially increases according to the following equation:

$$T_H=0.743\cdot e^{0,069\cdot\varepsilon} \qquad (R=0.99)$$

where ε [%] is the decreasing of the thickness of the sheet.

By using the comparative complete deformation (φ_t) the value of T_H can be calculated by the following equation:

$$T_H=1.29\cdot e^{3,21\cdot\varphi} \qquad (R=0.98)$$

I have found that during the cold rolling process the effect of microstructural changes to the T_H value is that:

- in case of cold rolling of EK2 type steels, the microcavities evolved by the fractures of the carbides play a determining role in the trapping of the hydrogen. The hydrogen traps (dislocations), which were evolved by cold rolling and can be eliminated by heat treatment, have perceptible effect if $\varepsilon > 30\%$ and ($\varphi_t > 0.41$);
- in case of cold rolling of EK2 type steels, till ~51% decreasing ($\varphi_t = 0.84$), the T_H value increases linearly with the ratio of (fragmented carbides)/(total carbides):

$$T_H = 0,23\cdot\frac{fragmented\ carbides}{total\ carbides}+0,22 \qquad (R=0.98)$$

Above this threshold limit, practically all of the carbides are broken.

- in case of cold rolling of EK2 type steels, till ε <60% (φ_t<1) between the T_H value and dislocation density (ρ) the following equation can be used:

$$T_H = 0,51\cdot e^{0,90\cdot\rho\cdot 10^{-14}} \qquad (R=0.97)$$

where the dislocation density [m^2] was determined by CMWP method.

I have concluded that in case of cold rolling of EK2 type steels, the evolving cracks in the carbides and on the ferrite/carbide phase boundaries penetrate only to those ferrite grains, which contact directly with the carbides.

(*Fábián, 2006b; Fábián, 2007; Fábián, 2008a; Fábián, 2010a; Fábián, 2010b; Fábián, Szabó, 2010, Fábián és társai, 2011)*

Thesis 3rd

I have determined that in case of the delivery state DC04EK and DC01EK type steels, as a result of the 850°C and 6 minutes long enamel firing followed by cooling, the ferrite-carbide microstructure became partially austenitic, and thereafter modifying into ferrite (96 ± 1%), with many fine (diameters are smaller than ~100 nm) precipitates and pearlite (4 ± 1%) with thin lamellar structure (~20-60 nm layers thickness). In the microstructure of the enamel fired samples a small amount of not-transformed carbide can be found, but microcavities can be detected as well, especially in the surroundings of the perlite colony.

The T_H value of the skin-pass rolled sheets after the firing for all cases complies the fish scale free enamelling condition by the MSZEN10209: 2000 standard(T_H >6.7 minutes). The long hydrogen permeation time at fired samples are ensured by the incoherent interface of the fine (less than 100 nm diameter) dispersed carbides with the ferritic matrix and by the increasing of the semicoherent interfaces between the ferrite and cementite layers with 20-60 nm thickness inside of the perlite.

(Fábián, 2004)

Thesis 4th

I have concluded that after cold deformations of DC04EK steel sheets the hydrogen permeation time is in connection with the pole density of ⟨111⟩ ∥ ND texture, with dislocation density and the type of dislocations. At DC04EK sheet with average hydrogen permeation time ~10 minute in case of the delivery state, in which microstructure the spherical carbides with 2-4μm^2 area are not necessarily on the ferritic grain boundaries and the volume of the microcavities is not significant, where after φ_t=0.21 cold rolling level (ε_z=15%) measured 12.7 minutes average T_H value, after φ_t=0.34 (ε_z=25%) deformation decreasing to less, than the half (6.2 minutes) the relationship betveen the T_H value and pole density of ⟨111⟩ ∥ ND texture and the dislocation densities are:

– between φ_t=0–0.56 (ε_z=0-40%) the T_H value increases linearly with the pole density volume of the (111) oriented rolled surface as the followings:

$$T_H=3.36\cdot x- 4.48 \qquad (R=0.76)$$

where x is the <111>∥NI orientated relative pole density of the s compared to the randomly oriented measurement situation.

– between φ_t=0.21–0.66 (ε_z=15-46%) deformation, near the <111>∥NI pole density adequate changing the dislocation density ρ [m^2] determined by X-ray diffraction measurements influences the T_H value as the followings:

$$T_H = 7.466 \ln(\rho 10^{-14}) + 7.74$$

The correlation coefficient: R = 0.87

– between φ_t=0,21–0,66 the observed minimum value of T_H is in connection with the increasing ratio of the screw dislocations. This was proven by the value of the connected point q=2,7 by CMWP method.

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