New continuous annealing cycle for producing DDQ steel sheets for automotive industries

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In automotive industry, one of the most important demands is the availability of high strength and deep lrawing quality steel sheets with excellent formability and good anti-aging properties. Formability is required when the sheet is shaped into an automobile body panel, and high strength is required after assembly. Recently, such steel sheets have been required to satisfy the need for car weight reduction and energy aving in the sheet coating process, i.e. lower coating temperature and shorter coating time, which contributes also to resolving global environmental issues. The application of Continuous Annealing Technology ıas allowed the production of DDQ steel sheets with bake hardening effect, which offer a good compromie between drawability and strength. Nevertheless, these steel sheets have poor anti-aging properties, vhich could be overcomed by controlling the solute carbon content. The most important factors influening the properties of the produced steel sheets by CAL are: carbon content, annealing temperature, slow ooling temperatures, rapid cooling and overaging conditions. The key point in the continuous annealing echnique is to decrease the solute carbon content up to a certain limit. This could be achieved by promoing the cementite precipitation and growth in the overaging stage. There is a big dilemma on the cementie precipitation site regarding the mechanical properties from one side and the anti-aging properties from he other side. Precipitating cementite in the ferrite matrix enhances the anti-aging properties however, reluces sheet formability. The optimum carbide precipitation necessary to strike a balance between ductility nd strain aging property is still a matter of discussion.

study was made on a new continuous annealing cycle (CAL) aims at improving the aging resistance and naintaining the compromise with formability characteristics.

Three different ELC steels with different carbon contents (0.02, 0.03, 0.05%) were industrially hot rolled, old rolled then continuously annealed on laboratory simulator. The continous annealing involved soaking, uenching from 625°C and overcooled to 250°C, retaining at the quenching temperature for 4.25 s, then ither reheating to 350°C, overaging for 180s, and cooled to 150°C, or reheating to 450°C, hot dip galvaizing and final cooling. The effect of carbon content and two-stage cooling on the aging characteristic nd mechanical properties of the steel sheets was studied.

Parole chiave: continuous annealing, DDQ, steel sheets, automotive, aging

INTRODUCTION

utomotive industry are recently required to conform to the ew global requirements regarding vehicle weight, energy onsumption and emission control. The new challenge to the eel makers for automotive industry, is to develop alternatie deep drawing steel products using advanced technology offer weight reduction, high mechanical properties and orrosion resistance.(1,2,3)

eep Drawing Quality steel sheets (DDQ) must have excelnt formability, good dent resistance and anti-aging propers. Formability is required when the steel sheet is shaped to automobile body panel, and high strength is required afr assembly.

he development of new and existing steels has led to new rmulations of bake hardening (BH) steels which can be oduced with formability comparable to that of the traditiol Interstitial Free (IF) grades, along with the ability to increase in strength once the parte has been formed and baked in a standard paint curing cycle. This combination of attributes allows thinner material to be used witout compromising strength. BH steels benefits initially from a relatively low dislocation density and a controlled level of carbon in solution. The strength achieved in BH steels is attributed to the interstitial carbon atoms migrating to and pinning dislocations. Once pinning has occured, an increased stress is required for further deformation.

Continuous evolution of steel producing technologies, specially the introduction of vaccum degassing technologies, paved the way to the production of extra-low carbon (ELC) steels, which is more resistant to aging at room temperature.

The technology of Continuous Annealing has long been used for the production of a variety of cold rolled steel sheets having the same quality as batch annealed products; such as hot-dip-galvanized sheets, tinplates, stainless-steel sheets, and nonoriented steel sheets.

The prime advantage of continuous annealing is the considerable increase in product uniformity, flatness, and surface cleanliness along the length of the given coil. This is very important as tolerences and allowable property variance are reduced as a result of the increasing automation of forming processes. Continuous annealing results also very economi-

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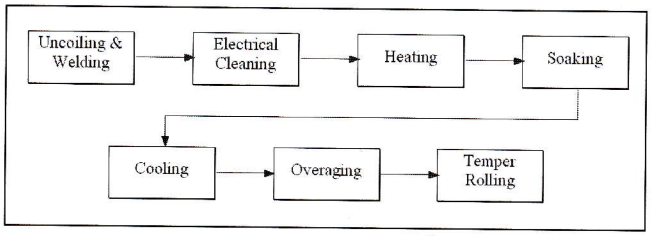

Fig. 1. Sequences of processes included in continuous annealing process line.

Fig. 1: Sequenza dei processi nella linea di ricottura continua

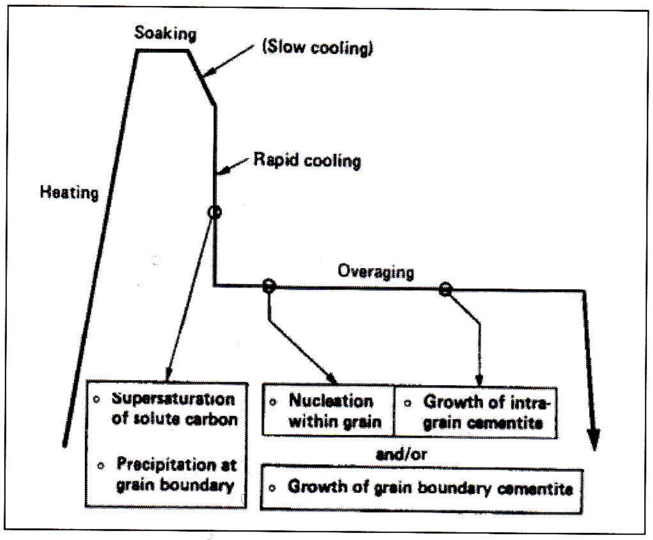

Fig. 2: Schematic illustration of continuous annealing cycle

Fig.2: Illustrazione schematica del ciclo di ricottura continua

cally attractive as it requires shorter processing time, low labor and handling costs, and improved yield resulting from the combination of several operations; electrolytical cleaning, annealing and temper rolling, as shown in Fig.1.(3,4)
Modern heat cycles of continuous annealing are divided into the following steps

- Rapid heating to a high temperature above A_1 (675°C - 850°C)
- Soaking for an annealing time on the order of 1 min.
- Slow cooling to below A_1 using a gas jet cooling (~ 10°C/s),
- Rapid cooling at rates 10-100°C/s
- Overaging at ~ 400°C for 1-5 minutes.
- Final cooling to room temperature.

Stages from rapid cooling to final cooling are particularly important as they determine the mechanical properties of the product. Texture and solute carbon content change in stages up to rapid cooling. During the overaging stage, the grain size and texture do not change at all, while a decrease in the solute carbon occurs. The key point in continuous annealing technique, for the production of DDQ steel sheets, is to reduce the solute carbon to a certain limit (< 10 ppm) by promoting the carbon precipitation as cementite and growth at the overaging step. Carbides are better recipitated at the grain boundaries than within the grains. The solute carbon is precipitated to improve the strain aging of the product, and the cementite growth is to secure the ductility.
Fig. 2 schematically shows the continuous annealing cycle along with the structural changes
Rapid cooling after soaking results in precipitation of very fine carbids inside the grains. In this case, strain aging property deteriorates, and high ductility is achieved. On the other hand, slow cooling gives the chance for coarse carbides precipitation on the grain boundaries and very few fine carbides are precipitated inside the grains. Consequently, we get good strain aging property and less detrioration of the ductility. Therefore, a certain compromise should be attained using a moderate cooling rate to satisfy both characteristics.
In automotive industries, the Aging Index is a very important measuring parameter to determine the soundness of steel type. Steels with aging index less than 20MPa are absolutely non-aging, and highly acceptable in all applications. Steels with A.I. between 20 and 40 MPa suffer some degree on aging and are suitable for limited applications. If the aging index is higher than 40MPa, steel is not acceptable for automotive industries.
A study was made on a new continuous annealing cycle (CAL) aims at improving the aging properties while maintaing the compromise with formability characteristics. A further improve in the absolute value of both strain aging and ductility, was thought to be obtained by increasing the driving force for carbide precipitation in the overaging stage. A high degree of supercooling is introduced in the CA cycle to further enhance the supersaturation of solute carbon before overaging.

Sample	C	Si	Mn	P	S	Al	N(ppm)
Fe-0.02C	0.02	0.01	0.14	0.009	0.007	0.039	33
Fe-0.03C	0.03	0.02	0.22	0.014	0.010	0.035	39
Fe-0.05C	0.05	0.01	0.23	0.015	0.019	0.042	38

Table 1. Detailed chemical composition (in weight percent) of the three Alloys

Tabella 1: Composizione chimica (wt%) delle tre leghe

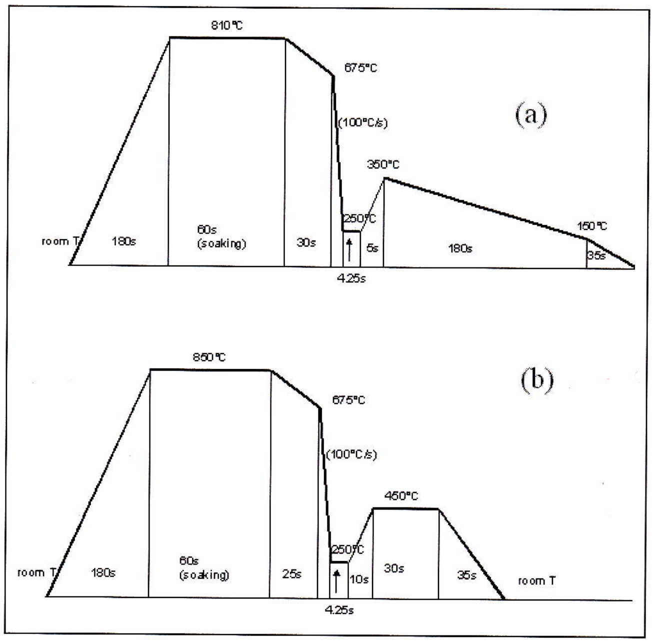

Fig. 3: Laboratoty simulation anealing cycles:
a) Continuous annealing, b) hot dip galvanizing

Fig.3: Cicli di simulazione delle ricotture in laboratorio
a) Ricottura continua, b) zincatura a caldo

EXPERIMENTAL PROCEDURE

Materials

Three different ELC steels with the chemical composition indicated in table 1., were industrially hot rolled at 650°C and cold rolled at 67%.

Heat Treatment

Samples were underwent two different laboratory heating regimes in Gleeble 3800. The first heating cycle simulates a continuous annealing line (Fig 3.a), which involves rapid heating to 810°C, soaking for 60s, slow cooling to 675°C in 30s, then quenched to 250°C at a rate of 100°C/s, reheating and overaging between 350°C and 150°C in 180s, then final cooling.

The second heat regime represents the hot dip galvanizing line (Fig 3.b), and is similar to the first program except that soaking is at 850°C for 60s, and galvanizing at 450°C for 30s.

Microscopy

The microstructure, grain size and cementite precipitation profile were observed with a Reichert MeF2 Optical microscope.

Mechanical Properties Measurements

The work hardening exponent (n-value) has generally been used for the estimation of the uniform elongation. It is determined by the dependance of the yield stress on the level of strain using the true stress-strain curve, according to the following equation:

$$\sigma_T = k\,\varepsilon^n \qquad (1)$$

Where k is a constant known as strength coefficient. The value of n was calculated in the deformation field of 10-20% elongation.

Plastic strain ratio ($\bar{r}$) relates the drawability, and is known as the anisotropy factor. It is defined as the ratio of the true width strain to the true thickness strain in the uniform elongation region of a tensile test.

$$\bar{r} = \varepsilon_w / \varepsilon_t \qquad (2)$$

Aging Index (A.I.)

Aging Index is the measure of the aging properties. It is calculated by the difference between the flow stress at 10% strain, and the lower yield strength after 10% straining and artificial aging (100°C, 60 min).

INFLUENCE OF CARBON CONTENT ON GRAIN SIZE AND MECHANICAL PROPERTIES

It is well knows that grain diameter is greatly influenced by carbon content.(5) As seen in Fig. 4, the grain diameter be-

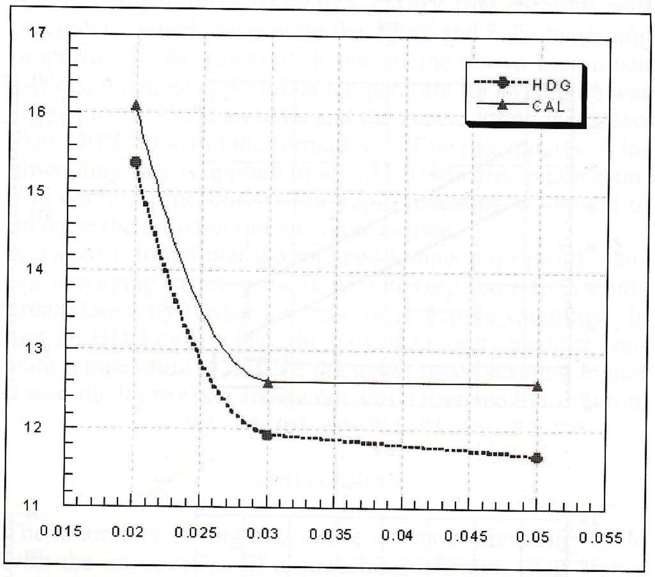

Fig.4: Effect of % carbon on the grain size

Fig. 4: Effetto del carbonio sulla dimensione del grano

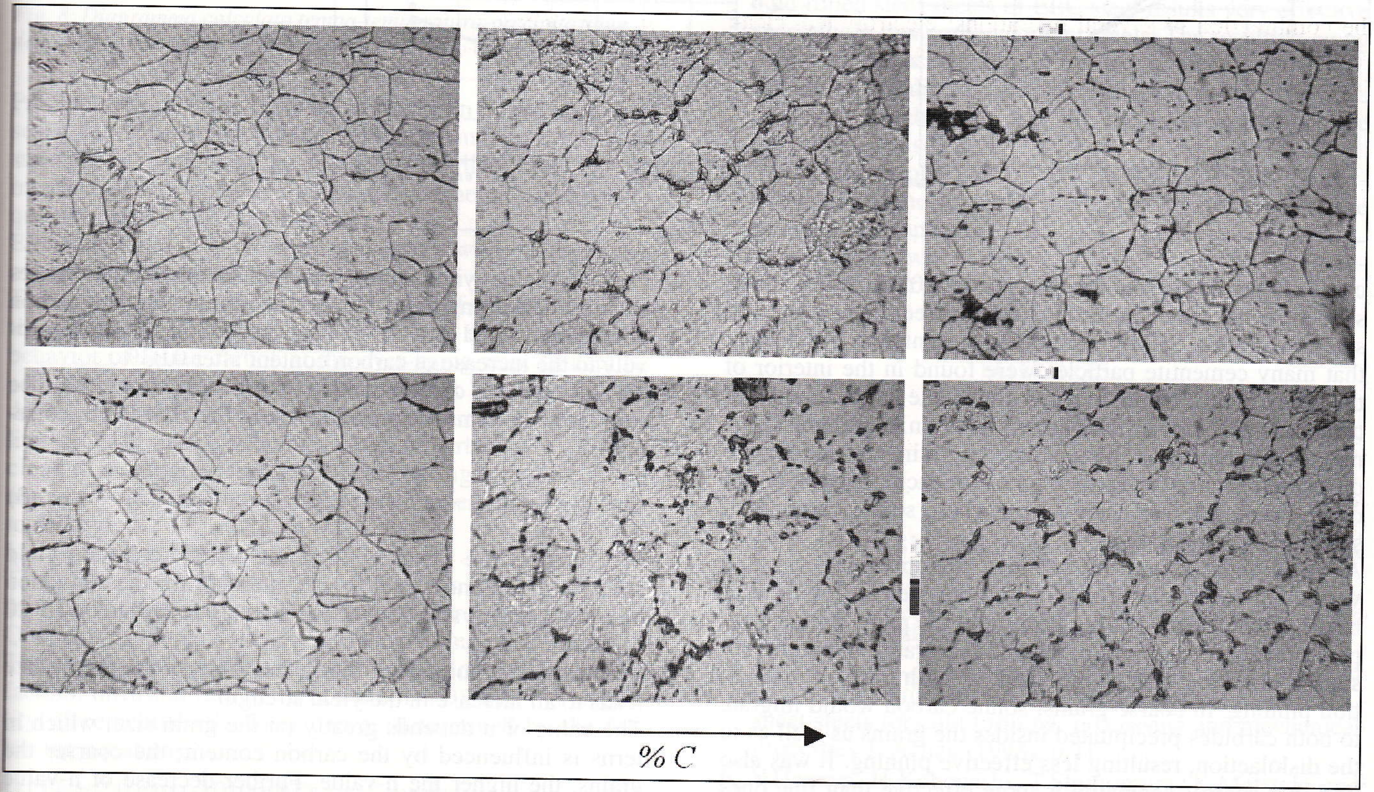

Fig. 5: SEM microgragh s for both CAL and HDG samples showing the effect of increasing carbon content of grain size and carbide precipitation site and dimension.(x500)

Fig. 5: Micrografie al SEM per il ciclo CAL e HDG; i campioni mostrano l'effetto dell'incremento del tenore di carbonio sui siti di precipitazione e le dimensioni dei carburi (X500)

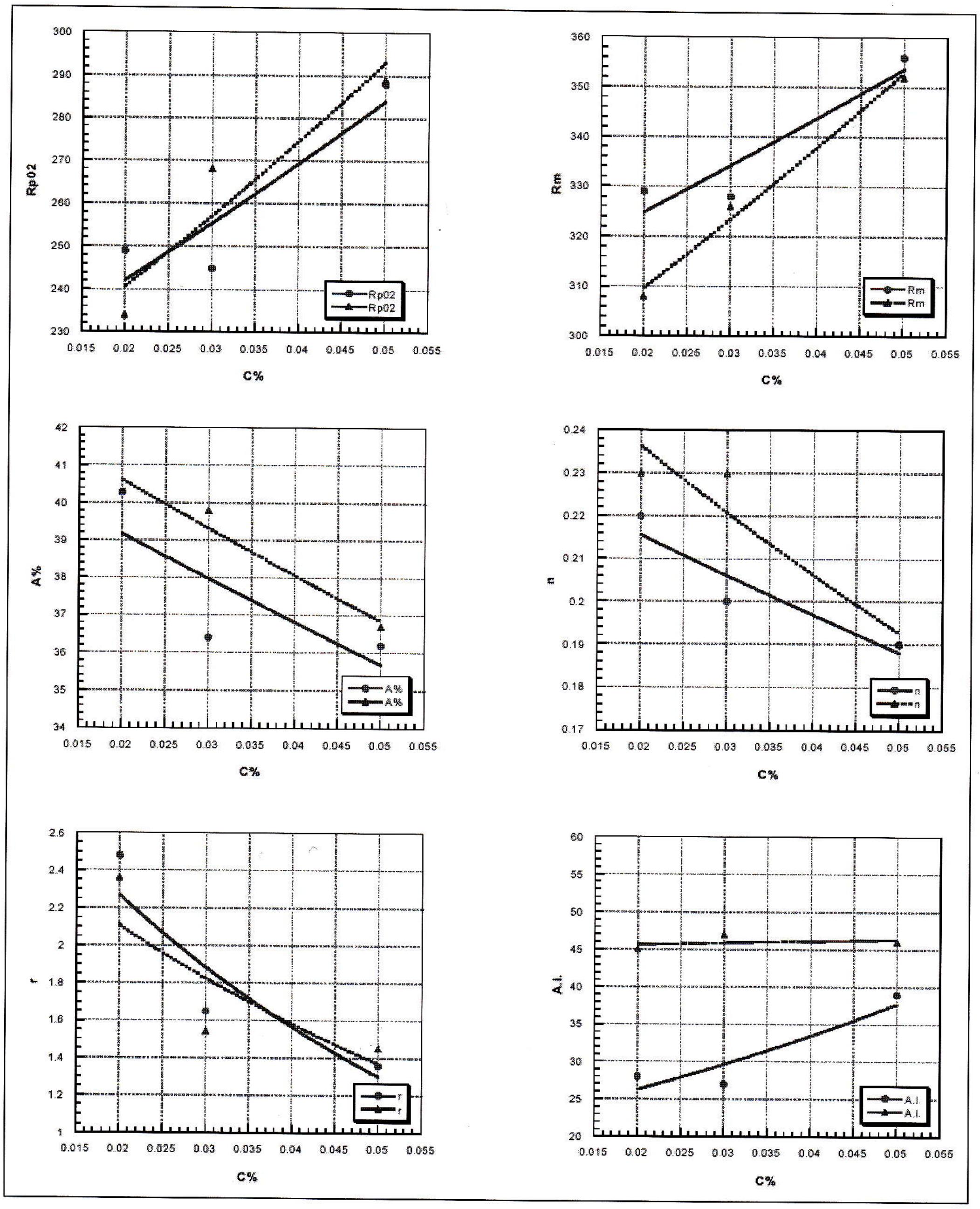

Fig. 6. Mechanical properties of both CAL and HDG

Fig.6: Proprietà meccaniche dei cicli CAL e HDG

comes larger as carbon decreases from 0.05 to 0.02%. Consequently, the grain size influences the precipitation site and size of carbides. SEM micrographs presented in Fig.5 show that many cementite particles were found in the interior of the larger grains; while, in small grains, the larger fraction of cementite particles was found on the grain boundaries. This may be explained as the smaller the grain, the shorter the diffusion distance for carbon atoms to reach the grain boundaries - being lower energy sites where solute carbon can store at low temperature.

As mentioned before, the carbides' precipitation sites play very significant role in controlling the yield strength after baking (BH effect). During heating to the paint baking temperature, solute carbon migrates to the grain boundaries resulting in an increase of the yield strength through dislocation pinning. In coarse grains, solute carbon would migrate to both carbides precipitated insides the grains as well as to the dislolaction, resulting less effective pinning. It was also find that coarser cementite is more effective than fine ones in dislocation pinning, as disclocations may cut the small particles.

Fig.6 shows the mechanical poperties of both CAL and HDG samples. It is found that with the increase of carbon content value, yield strength and tensile strength increases steadily. The aging index reaches its minimum when carbon content is around 0.03%. Elongation becomes more sensitively to the increase of carbon content after 0.03%.

The influence of carbon content on the yield strength can be expressed in terms of grain size using the Hall-Petch correlation:

$$\sigma_y = \sigma_i + k_y d^{-1/2} \qquad (3)$$

Where σ_y is the lower yield point (MPa), σ_i is the yield strength in a monocrystal, k_y is a measure for the activation of a dislocation source, for ELC-steels is typically 15-20 MPa/mm^2.

This equation points out that a decrease of the grain size leads to an increase in the yield strength.

The value of n depends greatly on the grain size, which in terns is influenced by the carbon content; the coarser the grains, the higher the n-value. Further decrease of n-value could be caused by the morphological and quantitative change in carbides. These phenomena are the product of the change in the precipitation site and the inter-carbide mean free path of fine carbides.

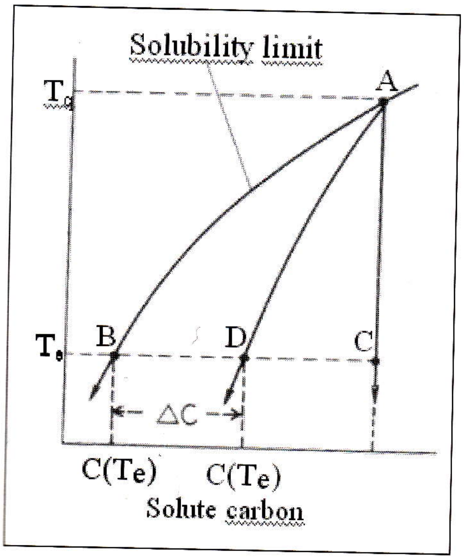

Fig. 7: Schematic illustration for the dependence of the carbon super- saturation degree on the cooling rate

Fig. 7: Dipendenza del grado di sovrassaturazion e del carbonio dalla velocità di raffreddamento

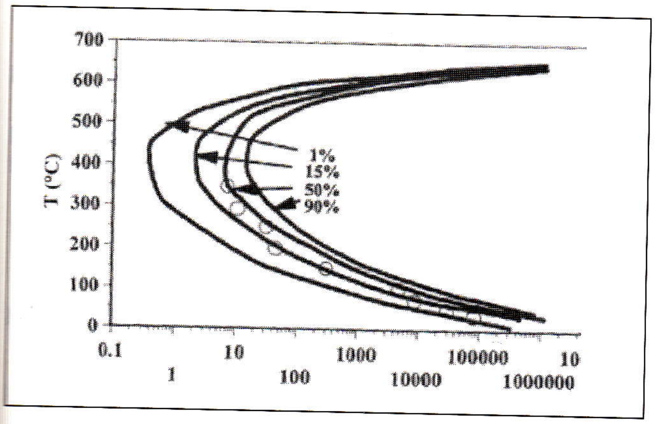

Fig. 8: Calculated time-temperature-precipitation diagram for cementite precipitation in ferrite containing 0.045%C.(7)

Fig. 8. Diagramma calcolato tempo-temperatura-precipitazione della Cementite in ferrite contenente lo 0.045% di carbonio.(7)

Precipitation of filmy carbides at the grain boundaries causes a decrease in the ferrite-cementite matrix interfacial strength; as it enhances the generation of micro-cracks at the interface of ferrite and grain boundary cementite, and consequently deteriorates ductility. (6)
Elongation and n-value decrease slightly with the reduction of cementite diameter.
The aging index is related to the amount of solute carbon after annealing, which in turn depends on the precipitation behavior of solute carbon at the over-aging step. Generally, carbide precipitation in overaging step is governed by the degree of supersaturation of solute carbon during rapid cooling (ΔC). From the solubility curve of carbon (Fig. 7), it is clear that under extremely high cooling rate (AC), the amount of supersaturated carbon at temperature T_e is estimated by:

$$\Delta C = C\,(T_e) - C\,(T_e)^* \qquad (4)$$

as a function of the quenching temperature (T_q), minimum Quenching Temperature (T_e) and the cooling rate.
The carbide precipitation site is a function of two competing factors; diffusion rate of carbon atoms (D), and the solubility of cementite (S). They can be determined by the following equations:

$$D\,(T) = 2.0 \exp(-10100/RT) \quad \mathrm{mm^2 s^{-1}} \qquad (5)$$
$$S\,(T) = 9.65 \exp(-12100/RT) \quad \mathrm{wt\%} \qquad (6)$$

The key point is to minimize the solubility of carbon and maximize the diffusion rate during overaging. The kinematical expression of carbide precipitation at the overaging step can be expressed as follows:

$$dC/dt = -k\,D(T)\,[C(T) - S(T)] \qquad (7)$$

Where k is a constant depends on the precipitation site and density.
To maximize the carbon precipitation at the overaging stage, a certain balance between the overaging temperature and time should be reached to maximize the term [C(T) – S(T)].
As presented in the time-temperature precipitation curve (Fig.8) for cementite in ferrite containing 0.045%C calculated by Koseschnik and Burchmayr(7), the highest precipitation yield can be achieved at around 350-450°C.
At low temperatures (90-250°C), very fine carbides will precipitate, which deteriorate the aging and bake hardening properties of the material. From all the above mentioned aspects, the most appropriate temperature for overaging was chosen to be 350°C in CAL and the supercooling is not less than 250°C to avoid the formation of fine precipitates. A high cooling rate is applied to avoid carbide precipitation during cooling procedure, while rapid reheating is applied to increase the difusion rate of solute carbon.
It was also found that the inclined temperature profile during overaging increase the (CAL) driving force for carbide precipitation by further decrease of cementite solubility. In fact, in HDG cyle, where the galvanization is made at constant temperature of 450°C, the aging index resulted higher due to the higher percentage of carbon remained in solution.

CONCLUSIONS

The laboratory testing of a new continuous annealing cycle with the introduction of a quenching-reheating step with a high degree of supercooling has led to the following conclusions:

- The use of the new annealing cycle for the production of cold rolled steel sheets of ELC steel results very effective specially in low carbon content steels. The resulting aging properties are acceptable for limited application.
- In case of hot dip galvanizing cycle, the aging properties are not acceptable, and it is still recommended to use the interstitial free steel.
- Mechanical properties are strictly dependant on the carbon content; the lower the carbon content the better the mechanical properties.

New heat cycles will be tested to further study the time-temperature effect for eventual industrial application.

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ABSTRACT

NUOVO CICLO DI RICOTTURA CONTINUA PER LA PRODUZIONE DI ACCIAI DDQ PER L'INDUSTRIA AUTOMOBILISTICA

Il fenomeno dell'invecchiamento degli acciai a basso carbonio da profondo stampaggio (Deep Drawing Quality) è particolarmente importante per la produzione di questi materiali nelle linee di ricottura continua e di zincatura. La precipitazione del carbonio durante le fasi di overaging dipende dalla composizione chimica dell'acciaio e dai parametri di ricottura e di raffreddamento. Il fenomeno viene particolarmente influenzato dalla sovrassaturazione di carbonio nella matrice ferritica all'inizio della fase di overaging. Proprio per aumentare il grado di sovrassaturazione, sono stati sperimentati cicli termici che prevedono il prolungamento del raffreddamento successivo al soaking fino a temperature inferiori a quelle di overaging, con successivo rapido riscaldo alla temperatura stessa di overaging. Con questi cicli sono stati ottenuti risultati interessanti per quanto riguarda soprattutto la simulazione della ricottura continua, dove gli acciai con più basso carbonio hanno mostrato accettabili valori di invecchiamento e buone caratteristiche meccaniche. Nel ciclo di zincatura invece, accanto a buoni valori di caratteristiche di stampabilità, non si sono associati valori accettabili dell'indice di invecchiamento a dimostrazione della presenza in questo ciclo di elevati valori del carbonio soluto nella ferrite.