

Ultrafine grained steels and the n coefficient of strain hardening

Aceros de grano ultrafino y el coeficiente n de endurecimiento por deformación

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Abstract.- Though many efforts have been made to obtain ultrafine grained steels, the industrial production level has not yet been reached due to the plastic instability during forming. The present work shows, using three examples, that high strength low alloy steels produced by advanced thermomechanical controlled rolling are in fact ultrafine grained steels with grain sizes below 5 μm , establishing also a connection between the coefficient of strain hardening, mechanical strength and admissible thickness tolerances in steel sheets, in order to use these materials in cold work forming (bending and drawing). A minimum value of coefficient n must be reached in order to assure commercial use of the steel sheet.

Key words: UFG steels, microstructure, mechanical properties, forming

Resumen.- Aunque se han hecho muchos esfuerzos para fabricar aceros de grano ultrafino, la producción nunca ha llegado a niveles industriales debido, sobre todo, a la inestabilidad plástica del material durante la deformación. A través de tres ejemplos, el presente trabajo muestra que los aceros de alta resistencia y baja aleación manufacturados por la técnica de rolado avanzado termomecánicamente controlado, son de hecho aceros ultrafinos con tamaños de grano por debajo de 5 μm , estableciendo además conexiones entre el coeficiente de endurecimiento por deformación, resistencia mecánica y tolerancia de los espesores admisibles en placas de acero, de forma que estos materiales puedan ser conformados en frío (plegado y estampado). El coeficiente n debe alcanzar un valor mínimo para asegurar el uso comercial de la placa de acero.

Palabras clave: aceros de grano ultrafino, microestructura, propiedades mecánicas, estampado.

1. Introducción.- Over the last years and in many countries (U.S., Great Britain, Germany, India, Japan and Australia) numerous researches have been carried out to obtain ultrafine grained steels (UGS). These investigations have not yet reached industrial level as the plastic instability during forming ($n \approx 0$) remains an unsolved problem. This work shows, using three examples, how high strength low alloy (HSLA) steels contemplated by Euronorm EN 10149-2 standard and manufactured by advanced thermo-mechanical controlled rolling processes (ATMCRP) are in fact UFG steels and are used primarily in the automotive and construction sectors. Following well established research works and thickness tolerances industrially admitted in hot work

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lamination, it is shown that values above $n=0.1$ are indispensable for UFG steels to be subsequently cold-formed.

In recent years, both the steelmaking industry and laboratories in different parts of the world, have shown an increasing interest in reaching an industrial level production of ultrafine grained steels (ultrafine ferrite – UFF, ultrafine grain – UFG)¹, being the most interesting ones those with grain size d (mean linear intercept) lower than 5 μm (grain size G ASTM higher than 12), resulting in an increase in mechanical resistance and fracture toughness expected from these microstructures². Though, till present time, none of the different techniques that have been set up at a laboratory level, such as severe plastic deformation (SPD)³⁻⁵, accumulative roll bonding (ARB)⁶, dynamic strain induced deformation (DSIT)^{7,8} and rapid transformation annealing (RTA)⁹, have achieved to overcome the plastic instability consequence of the low strain hardening n coefficient value that the UFG steels manufactured by these procedures show¹⁰.

Only advanced techniques of control rolling such as advanced thermomechanical cold rolling processes (ATMCRP) used by the steelmaking industry in collaboration with research labs such as the Sid-met-mat of ETSIMO (Oviedo University, Spain), have obtained industrial level productions of UFG steels. This is the case of the Arcelor Mittal de Asturias Group (Spain), whose UFF steel productions in raw hot rolled state overpassed the 60,000T level in 2010, in the form of hot rolled sheet (thickness above 1.5 mm). This work tries to establish, in a resumed form, the connection between the n coefficient, grain size, mechanical resistance and thickness tolerances admissible in steel sheets, so that UFG steels may be used in cold work forming operations for bending and drawing.

2. Experimental Work and Results.- Data from 3 UFG steels are presented, with code names S355, S420 and S500, contained in the Euronorm EN10149-2 standard (Specification for hot-rolled flat products made of high yield strength steels for cold forming. Delivery conditions for thermomechanically rolled steels), whose chemical composition and mechanical properties are specified in table I and table II respectively. The steel was delivered in the form of steel sheet with a thickness equal or below 8 mm and were produced by ATMCRP (hot rolled raw state) in the industrial facility of Arcelor Mittal de Avilés (Asturias, Spain). These materials are primarily used in the automotive sector (reinforcements and supports) and in the construction sector (ties, angles and crane arms). Table III shows the chemical analysis, obtained by the average of 3 samples of the steel sheets investigated. Steel S355 and S420 are microalloyed with Nb and low Mn. The S500 steel is microalloyed with Nb-Ti and high Mn, all of them within the specified composition required by the Euronorm.

Name	Material number	C% max	Mn% max	Si% max	P% max	S% max**	Al total % min	Nb% max*	V% max*	Ti% max*	Mo% max	B% max
S315	1.0972	0.12	1.30	0.50	0.025	0.020	0.015	0.09	0.20	0.15	--	--
S355	1.0976	0.12	1.50	0.50	0.025	0.020	0.015	0.09	0.20	0.15	--	--
S420	1.0980	0.12	1.60	0.50	0.025	0.015	0.015	0.09	0.20	0.15	--	--
S460	1.0982	0.12	1.60	0.50	0.025	0.015	0.015	0.09	0.20	0.15	--	--
S500	1.0984	0.12	1.70	0.50	0.025	0.015	0.015	0.09	0.20	0.15	--	--
S550	1.0986	0.12	1.80	0.50	0.025	0.015	0.015	0.09	0.20	0.15	--	--
S600	1.8969	0.12	1.90	0.50	0.025	0.015	0.015	0.09	0.20	0.22	0.50	0.005
S650	1.8976	0.12	2.00	0.60	0.025	0.015	0.015	0.09	0.20	0.22	0.50	0.005
S700	1.8974	0.12	2.10	0.60	0.025	0.015	0.015	0.09	0.20	0.22	0.50	0.005

Table I: Chemical composition of thermomechanically rolled steels. *The sum of Nb, V and Ti shall be max. 0.22%. **If ordered, the sulphur content shall be max. 0.01%

Name	Material number	Minimum yield strength [MPa]	Tensile strength [MPa]	Min percentage elongation at fracture		Bending at 180° min. mandrel diameter
				t<3 L ₀ =80 mm	t≥3 L ₀ =5.65√S ₀	
S315	1.0972	315	390 – 510	20	24	0t
S355	1.0976	355	430 – 550	19	23	0.5t
S420	1.0980	420	480 – 620	16	19	0.5t
S460	1.0982	460	520 – 670	14	17	1t
S500	1.0984	500	550 – 700	12	14	1t
S550	1.0986	550	600 – 760	12	14	1.5t
S600	1.8969	600	650 – 820	11	13	1.5t
S650	1.8976	650	700 – 880	10	12	2t
S700	1.8974	700	750 - 950	10	12	2t

Table II: Mechanical properties for thermomechanically rolled steels. The values for the tensile test apply to longitudinal test pieces. The values for the bent test apply to transverse test pieces. For t>8 mm the min. yield strength can be 20 MPa lower.

Sample	S355	S420	S500
C	0.09	0.10	0.09
Mn	0.35	0.47	1.41
Si	0.02	0.01	0.02
S	0.008	0.010	0.012
P	0.011	0.016	0.019
Ti	--	--	0.075
Nb	0.021	0.044	0.047
Al	0.038	0.029	0.031

Table III: Chemical analysis of the three steel samples tested.

Table IV presents the mechanical characteristics of the steel sheets tested parallel to the rolling direction, according to EN1002-1. The value of the n coefficient was calculated from the engineering curves, as indicated by ASTM E-646-00 (Standard Test Method for Tensile Strain-Hardening Exponents “ n -Values” of Metallic Sheet Materials) which allows the calculation of the rational tension curve $\sigma = k\varepsilon^n$ (σ true stress; ε true strain; k resistance coefficient). Figure 1 shows the engineering stress-strain curve (s-e) of the mentioned steel sheets, adding as a reference the curves of 2 other cold worked steels: an IF steel (free of interstitials and microalloyed with Ti) and a dual-phase DP (Si-Mn-B) steel also produced at Arcelor Mittal in Asturias¹¹. Figure 2 presents for the same steels the logarithmic scale of both stress (σ) and strain (ε) which is used to calculate n and k coefficients by linear regression adjustments of the data.

Sample	S355	S420	S500
t (mm)	3.5	5.2	4.5
R _{eH} (MPa)	401	465	630
R _{eL} (MPa)	390	449	618
e _L (%)	1.6	2.0	2.3
R _m (MPa)	440	534	678
A (%)	33*	27**	26*
R _{eL} /R _m	0.89	0.84	0.91
n	0.12	0.16	0.10
\bar{G} (ASTM)	13.2	13.3	14.2
σ_G (ASTM)	1.40	1.47	1.05
\bar{L} (μ m)	3.2	3.1	2.3
%RA	7.6	7.9	5.3

Table IV: Mechanical properties and parameters of the samples tested, along with quantitative metallographic results. *L0=50 mm. ** L0=55 mm

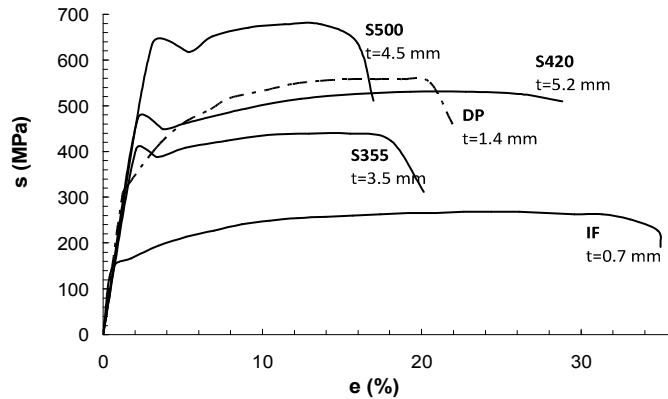


Figure 1: Stress-strain engineering curves of investigated steel sheets and data of DP and IF [11]

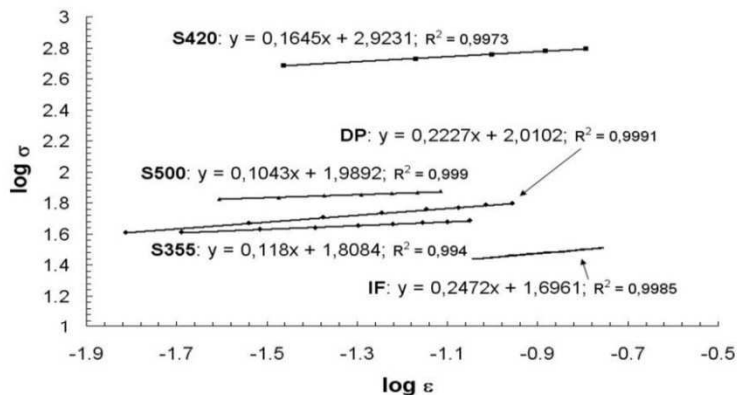


Figure 2: Linear regression equations from the logarithmic stress-strain plot to determine n coefficient

To determine the grain size G and mean linear intercept \bar{L} , metallographic samples of the steel sheets were obtained from the transverse section of the orientation parallel to the rolling

direction, they were subsequently polished and etched with Nital-2 to reveal the microstructure. Measurements were made in an automatic digital image analysis equipment coupled to a metallographic optical microscope, operated according to ASTM E-112 and ASTM E-1181-02 standards. Counting contemplated as many micrographs as necessary to obtain mean values of G and \bar{L} , with a confidence level interval of 95 per cent and a deviation lower than 10 per cent¹². Table IV also summarizes mean values of G , standard deviation σ , mean linear intercept \bar{L} and mean deviation RA. The relation between G and \bar{L} was obtained using the formula¹³:

$$G = -3.356 - 6.644 \log \bar{L} (mm) \quad [1]$$

Figures 3, 4 and 5 show micrographs along with counting patterns and histograms of grain number G , that were used to determine the data of table IV of the investigated steels. As expected, these are UFG steels with a structure majorly ferritic and a small amount (less than 10 per cent) of pearlite.

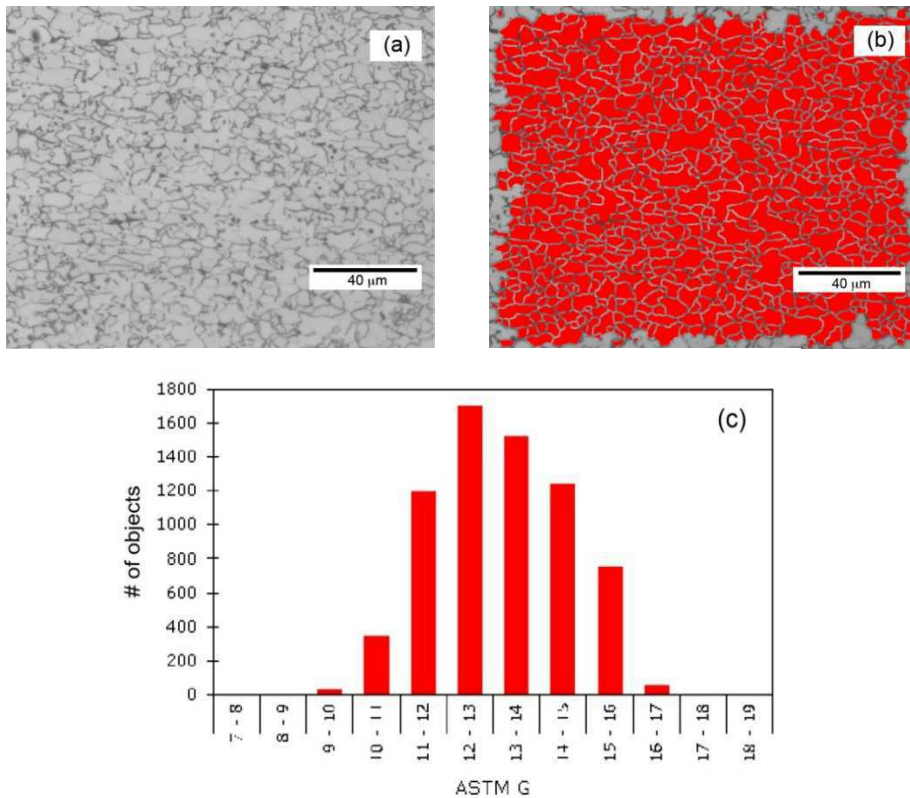


Figure 3: Metallographic image of sample S355 (a), along with detected grain pattern (b) and ASTM G grain size distribution histogram (c)

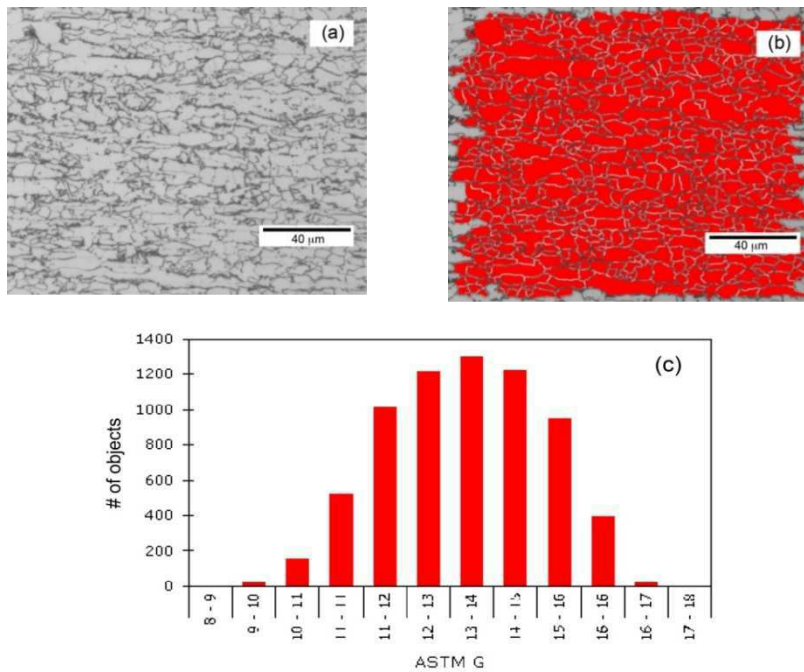


Figure 4: Metallographic image of sample S420 (a), along with detected grain pattern (b) and ASTM G grain size distribution histogram (c)

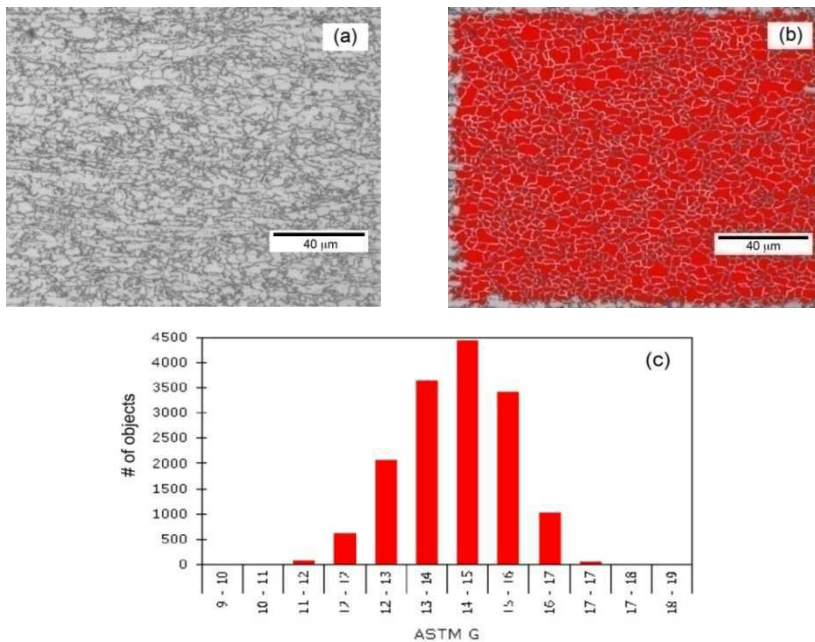


Figure 5: Metallographic image of sample S500 (a), along with detected grain pattern (b) and ASTM G grain size distribution histogram (c)

3. Discussion.- It is a well known fact in physical metallurgy, the plastic instability that the UFG steels may present when tension tested¹⁴⁻²⁰. In the case of this work, as shown in fig. 1, the yielding instability zones of the steels are between 1.5 and 2.5 per cent, but after that, a uniform plastic strain-hardening deformation is followed. There are three reasons for this:

3.1 Strain-hardening n coefficient of these steels diminishes with grain size, in accordance to a Morrison type law^[14]:

$$n = 5 / (10 + d^{-1/2}) \quad [2]$$

where d is the mean linear intercept (\bar{L}) in mm. Thus, in a steel with grain size $d=10\mu\text{m}$ (0.010 mm), the theoretical n value would be $n=0.25$; for a grain size of $d=1\mu\text{m}$, the coefficient would be $n=0.12$, which are calculations that usually exceed the real n value.

3.2 The second reason, based on the first one, explains the increasing plastic instability in UFG steels as the grain size becomes smaller. Heterogeneous deformations are generated in the steel, known as Lüders bands, with increasing amplitude ε_L as the capacity of the material for strain-hardening diminishes. When $\varepsilon_L \approx n$ the steel presents necking without showing uniform plastic deformation.

Morrison and Miller¹⁵ quantified the plastic instability limit condition for a steel with a true stress-strain curve that must fit a Ludwik-Hollomon expression of the type $\sigma = k\varepsilon^n$. In other words:

$$\varepsilon_L = n \quad [3]$$

If s_y is designated as the lower engineering yield stress in tension:

$$\sigma_y = s_y (1 + e_L) = k\varepsilon_L^n \quad [4]$$

where σ_y is the true tension yield stress. Combining equations [3] and [4]:

$$k/s_y = \exp[n(1 - \ln \varepsilon_L)] \quad [5]$$

for which the limit curve ($\varepsilon_L = n$) is represented in figure 6 as k/s_y versus n . This last figure highlights that all the steels with traction parameters below the limit curve will not reach uniform plastic deformation levels. The steels presented in this work are above the limit curve but very close to it ($n \approx 0.1$). On the contrary, IF and DP steels, taken as a reference, are well above the limit curve and generate high uniform plastic deformations as they show n values above 0.2.

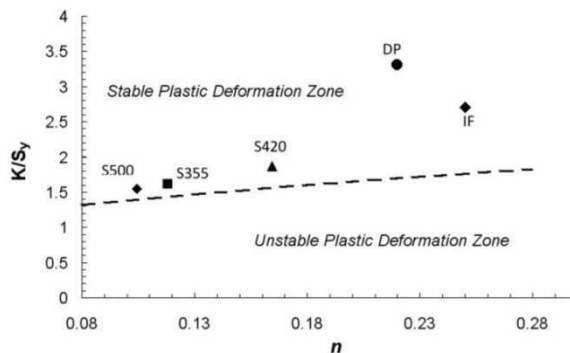


Figure 6: K/S_y ratio plotted vs. n coefficient for the investigated steel sheets

3.3 Cold work in these steels, both from forward deep drawing, as by biaxial expansion is possible when the sheets (mechanically considered as membranes with plane stress states) have strain hardening n coefficients and thickness sufficiently high enough. Keeler-Goodwin^{21,22},

Backofen¹⁵ and Hosford¹⁹ have shown, among others, that the critical state of deformation in a stamping process is the plane strain state: ε_1 (maximum principal strain in the plane of the sheet) = n ; ε_2 (minimum principal strain in the plane of the sheet) = 0; strain according to thickness $\varepsilon_3 = -\varepsilon_1 = n$ ²³. As these formulas show, the n coefficient is a critical factor. Furthermore, in biaxial forward stretching, in order for the sheet to access forming in plane-stress conditions without necking (deformation is located in the thickness direction), it is only possible if the sheet has weaker zones or less thick ones, that allow the plastic collapse. Because of that, Marciniak and Kuczynski²⁴ defined a defect factor $f = t_{0B}/t_{0A}$ (figure 7) which would make the necking possible in trajectories $\sigma_2/\sigma_1 > 0$ (where σ_2 and σ_1 are respectively the minimum and maximum biaxial tensions at the plane of the sheet). In these conditions the maximum load the sheet can withstand is reached when the deformation at the defect equals the n value. Therefore:

$$\varepsilon_1 > 0; \varepsilon_2 = 0; \varepsilon_3 = -\varepsilon_1 \text{ (plane strain)} \quad [6]$$

$$\ln(t/t_0) = -\ln(l_1/l_0) = -n \text{ (at the limit); } t = t_0 \exp(-n) \quad [7]$$

$$\sigma_{1A} t_A = \sigma_{1B} t_B \text{ (stress compatibility)} \quad [8]$$

$$k \varepsilon_A^n t_{0A} \exp(-\varepsilon_A) = k \varepsilon_B^n t_{0B} \exp(-n) = k n^n t_{0B} \exp(-n) \quad [9]$$

$$\varepsilon_A^n \exp(-\varepsilon_A) = (t_{0B}/t_{0A}) n^n \exp(-n) = f n^n \exp(-n) \quad [10]$$

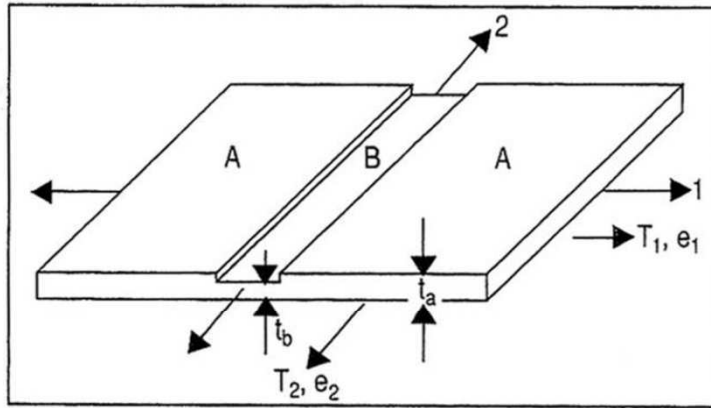


Figure 7: Schematic representation of a steel sheet with preexisting defect. Plane strain conditions

An approximate solution to equation [8] is the following:

$$\varepsilon_A = \varepsilon_u = n - \sqrt{n(1-f)} \quad [11]$$

Figure 8 presents the variation of ε_A (limit deformation) as a function of the defect factor $1-f$ for values of $n = 0.05, 0.15$ and 0.25 respectively. It may be observed that for thickness tolerances of $f \approx 0.990$, inferior to the ones admitted in the Euronorm EN 10051:199 (Continuously hot-rolled uncoated plate, sheet and strip of non-alloy and alloy steels. Tolerances on dimensions and shape) in hot rolled products ($f \approx 0.975$), and superior to cold ones ($f \approx 0.995$), with values of $n \approx 0.1$ the sheet may present necking, as if it were cold-worked rolled before withstanding substantial homogeneous deformations. Thus, if the f factor and thickness tolerances of the sheet

are added to the decrease in the n value as the grain size diminishes (UFG steels), cold work forming in biaxial expansion will not withstand homogeneous deformations as required by the manufacturing process.

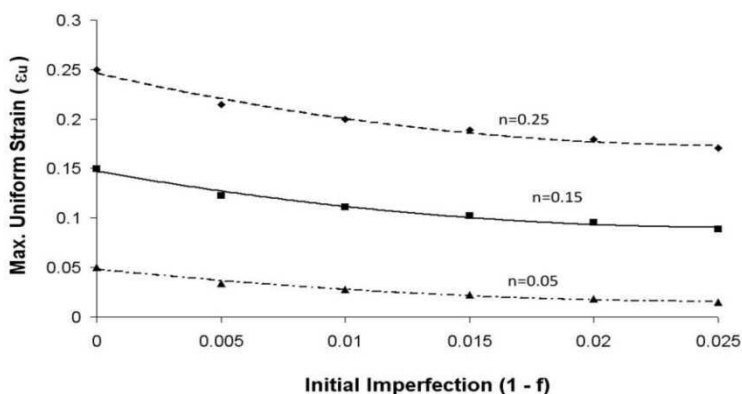


Figure 8: Maximum uniform strain as a function of initial imperfection $1-f$ in the steel sheet for n coefficient values of 0.05, 0.15 and 0.25

4. Conclusions.- Steels standardized by Euronorm EN-10149-2 in its raw state of hot rolling are in fact UFG (UFG) steels, showing yield stresses in the interval of 315 to 700 MPa and strain hardening coefficients that decrease with yield stress but superior to 0.1.

There are two factors that result in the extinction of the n coefficient, that disable cold-work operations. The first one is an excessively small grain size close to $1\mu\text{m}$ (16-17 ASTM), which makes uniform deformation processes and strain hardening impossible.

The second one is that thickness tolerances, necessarily admitted in hot and cold rolled products ($0.975 < f < 0.995$) have an impact in the form of defects in products manufactured by biaxial drawing, so for values of n lower than 0.1 (which means that maximum homogeneous deformations ϵ_u reaches 0) the steel may not be cold formed.

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