## LOM Characterization of Heat-Resistant Cast Steels for the Lime/Cement Industry

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Heat-resistant cast steels are highly alloyed stainless steels designed to withstand high temperatures (600-1000°C), in corrosive environments. They are nickel-chromium-iron alloys usually with ~0.2-0.75% carbon exhibiting a high strength at the elevated temperatures at which they operate [1]. The selection criteria for successful in-service operation include: corrosion resistance (oxidation, carburizing, nitriding and sulfidation); thermal fatigue and thermal shock resistance dependant on physical properties such as the lineal expansion coefficient,  $\alpha$ , and the thermal conductivity,  $\kappa$ ; metallurgical stability (mainly based in the ability to retard the formation of  $\sigma$ - phase); and lastly to exhibit good mechanical properties ordinarily tested at RT for simplicity by means of evaluation of harness and tensile testing [2].

In the present work three feeder links of a limestone continuous precalcining Lepol unit, operating between 300 and 750 °C (572-1382 °F) over one cycle, and with a thermal shock between 300-500 °C (572-932 °F), the last one owed to construction modifications derived from the adaptation of this roasting unit from cement to lime production. The advent of the present economical crisis has pushed manufacturers to the usage of cheaper fossil fuels, however, rich in sulphur (petroleum coke of ~ 2% S) mixed with natural gas, and due to the low specification in sulphur of the steelmaking lime it is manufactured (S<0.040%-wt.), which in turn obligated setting the temperature at higher values to avoid sulphur pick-up near the gas exit section.

The studied links were manufactured in accordance to DIN GX40 Cr Ni Si 22/9, the Cr-Ni-Si group designed for parts with a maximum operational temperature of 950°C, fair resistance to  $S^{2-}$ , good thermal shock resistance, apt for parts subjected to high loads at oscillating temperatures. As-cast microstructure corresponds to refractory austenitic stainless steels, due to the gamma forming character of 9%Ni and 0.4%C which prevails over the alpha forming one given by 22% Cr and ~1.5% Si. Carbon in the supply condition is usually distributed in equal parts, as interdendritic carbides on one side, and in solid solution of  $\gamma$  on the other. The links were set aside after a 5 year campaign and were identified as: sample A or "standardized" because it met all composition values in DIN standard; sample B with lower Ni than the value set in the specification, but with 0.5% Mo and 0.15% Nb added (not in the standard), both alpha and carbide forming elements. And lastly sample C, with a deficit in Cr and Ni with respect to the standard (Table 1). Samples for metallography were cut from a new link taken as a reference material (22% Cr-10% Ni), and of the three experimental compositions. The samples were mechanically ground and polished and etched with several etchants to reveal in light optical microscopy the relevant features to evaluate by quantitative metallography, namely the grain boundary carbides, grain carbides and  $\sigma$  phase (Fig. 1). The analysis revealed that damage by porosity was bigger in sample C (low Ni & low Cr) a fact which was coincident with high values of both the volume fraction of  $\sigma$  phase and gb carbides (M<sub>23</sub>C<sub>6</sub>) due to C precipitation and coarsening leading to gb decohesion. Sample B (Mo+Nb, low Ni), with the lowest  $\gamma$  grain size, displayed an intermediate fraction of porosity. Standardized sample A exhibited the lowest degree of intergranular porosity, coupled to low fractions of inter- and intragranular carbides (Figure 2).

## References:

[1] "High-Alloy Cast Steels" in ASM Specialty Handbook: Heat-Resistant Materials, ed., J. R. Davis, ASM International, Materials Park OH, p. 200.

[2] F.E. White and T.H. White, Traitement Thermique Revue **120-77** (1977), p. 43.

[3] The authors acknowledge the technical staff of Cementos Tudela-Veguin, S.A. for allowing the access to their factory, survey of the experimental material and for valuable technical discussions.

Element	DIN Standard	Sample A	Sample B	Sample C
	GX40 Cr Ni Si 22/9	"Standardized"	"Mo+Nb added"	"low-Ni & low-Cr"
С	0.30 - 0.50	0.34	0.32	0.31
Si	1 - 2.5	1.353	1.580	1.533
Mn	≤ 1.5	0.593	0.580	0.752
Cr	21 - 23	21.099	21.938	<u>20.243</u>
Ni	9 - 11	10.43	<u>8.25</u>	<u>7.81</u>
Mo		0.128	0.488	0.216
V		0.0882	0.0791	0.0627
Nb		0.0478	<u>0.1346</u>	0.0156
Р	$\leq 0.045$	0.030	0.031	0.030
S	≤ 0.030	0.023	0.032	0.024

Table 1. Compositional range for DIN Standard and corresponding values for samples A- C. (%-wt.).



**Figure 1.** LOM of samples A (left), B (intermediate) and C (right). Etched with: 2.5g FeCl<sub>3</sub>, 5g picric acid, 90 ml ethanol, immerse for  $\sim$ 15 s. Scale bar displayed at the bottom right: 100µm.



**Figure 2.** Quantitative metallographic results for the three studied samples: (a) volume fraction of porosity or damage (left); and (b) volume fraction of sigma phase (right) after 5 years in operation.