

Tema: Metalurgia Física

# EFFECT OF COLD ROLLING ON TEXTURE, ANISOTROPY AND MICROSTRUCTURE OF ANNEALED MARAGING 350 STEEL\*

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#### Resumo

Os aços maraging tem grande importância tecnológica devido sua aplicação nas industrias aeroespacial e nuclear. Uma das aplicações para esse material é a fabricação de cilindros de alta resistência pelo processo de estampagem profunda, logo o conhecimento da textura cristalográfica torna-se fundamental. Este trabalho investigou o efeito da laminação à frio seguida por um recozimento sobre a microestrutura, textura e anisotropia plástica no aço maraging 350. A anisotropia foi medida pelo cálculo dos coeficientes de Lankford e dos coeficientes de anisotropia normal (Rm) e planar ( $\Delta$ R). Os resultados mostraram o aumento na deformação a frio gera um aumento na intensidade da fibra <111> acompanhada de um aumento no valor de Rm. As análises metalográficas mostram que maiores reduções induzem à um precipitação intermetálica após o recozimento.

Palavras-chave: Maraging 350; Textura; Anisotropia

#### EFFECT OF COLD ROLLING ON TEXTURE, ANISOTROPY AND MICROSTRUCTURE OF ANNEALED MARAGING 350 STEEL

#### Abstract

Maraging steels have a great technological importance due their application in the aerospace and nuclear industries. One of these applications is a deep drawing for a fabricate cylinders of high strength, so knowing the mechanical behavior of this steel is critical to a good drawing. This paper evaluates the texture, anisotropy and the microstructure of maraging 350 steel after cold rolling and annealing. The anisotropy was measured by the calculation of Lankford's coefficients and the calculation the normal (Rm) and planar ( $\Delta$ R) anisotropy coefficients. It was found that increasing cold rolling reduction before the annealing strengths the <111> fibre texture and improves the Rm. The microstructural analysis shows that great rolling reduction could induce intermetallic precipitation after the annealing in this steel. **Keywords:** Maraging 350; Texture; Anisotropy.

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# 1 INTRODUÇÃO

Maraging steels are Fe-Ni-Co-Mo-Ti alloys with a low carbon content, that have a great application in aerospace, war and nuclear industries. Because it is soft martensite in the annealed condition this material shows a high plasticity in contrast with the high mechanical resistance in aged condition. In this steel the main hardener alloying element is the Ti, controlling the amount of Ni<sub>3</sub>Ti formed during the aging [1]. According to Abreu [2], the cold deformation of a polycrystalline material can induce the formation of a preferential orientation on grains, leading to a deformation crystallographic texture. Hosoya et. Al [3] proved that the recrystallization texture on maraging steels is similar to the other cold deformed BCC metals, transmitting to annealed condition a strong  $\gamma$  fiber and other paper [4] show that is possible to find the cube on edge component. Abreu [4] shows that the a deformation texture is strengthened by aging. Figure 1 shows sections of  $\phi2=45^\circ$  of the ODFs for some conditions of maraging 350 steel.



**Figure 1.** Sections of  $\varphi 2=45^{\circ}$  for the ODFs of maraging 350 steel in a) hot rolled condition to 60% of thickness, b) Cold rolling with a reduction of 80% in thickness and, c) Annealed to 820°C for 1h.

The crystallographic texture has strong influence on material's properties anisotropy. In the papers, there is no information about the anisotropy behavior of maraging steels in annealed condition.

One method to measure the plasticity anisotropy of a material is determining the Lankford's coefficients defined by the Eq.1

$$R = \epsilon_y / \epsilon_z$$
 (1)

Were  $\varepsilon_y$  is the true deformation on normal direction and  $\varepsilon_z$  is the true deformation on transverse direction in a tensile test. With the R<sub>0</sub>, R<sub>45</sub> and R<sub>90</sub>, the Rm (planar anisotropy coefficient), defined by Eq.2, and  $\Delta R$  (normal anisotropy coefficient), defined by Eq.2, and  $\Delta R$  (normal anisotropy coefficient), defined by Eq.3, can be calculated.

$$Rm = (R0 + 2*R45 + R90) / 4$$
 (2)

$$\Delta R = (R0 - 2^*R45 + R90)/2 \quad (3)$$

In a good material for deep drawing the values of Rm are higher than 1 to avoid a excessive wall thinning during the drawing [5], and a desired value  $\Delta R$  is close to zero to avoid the formation of ears after the drawing [6]. In the Figure 2, the effect of  $\Delta R$  can be observed in the drawing of a cup [7].

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**Figure 2.** Effect of  $\Delta R$  parameter on deep drawing.

## **2 EXPERIMENTAL PROCEDURE**

The chemical composition of the maraging steel used on this paper is shown on the Table 1. This steel was provided by Brazilian Navy in forged condition. The received steel was initially annealed on 900°C for 1h and after this was cold rolled in a LE 200 B roller provided by SENAI DR-CE, on Maracanaú Industrial District. The reduction by cold rolling was performed to reduction on 50%, 70% and 80% on thickness reduction and after the rolling the samples was annealed in a muffle furnace EDG 6P at 900°C for 1h. The identity of the samples is shown on Table 2.

Table 1. Chemical composition of Maraging 350 steel

Steel	Ni	Со	Mo	Ti	AI	Fe
Mar350	18,19	12,17	4,85	1,38	0,28	Bal

Table 2. Identification of Samples								
	Deformation	Colling	Identity of sample					
	Cold Rolled 50 %	Coll. Furnace	M350L50F					
	Cold Rolled 70 %	Coll. Furnace	M350L70F					
	Cold Rolled 80%	Coll. Furnace	M350L80F					

The samples used to measure the pole figures were corroded with a acid solution [40 ml H2SO4 + 40 ml H2O + 20 ml HNO3 ] to avoid the gridding damage to surface. A Phillips X'Pert diffraction system equipped with a Co target tube was used to collect data from texture analysis. A optical microscope Olympus BX51M and a SEM XL30 was used for perform the microstructural analysis. The samples used in metallography was etched with modified ferrous chloride [2,5g FeCl3.6H2O + 20 ml HNO3 + 40 ml HCl + 40 ml H2O] for 20s [8].

The calculation of the ODF performed with the POpla software and the sections of  $\varphi 2=45^{\circ}$  was plotted with the MTEX software package.

The tensile tests for calculation of Lankford coefficients was performed on a EMIC DL 10000 using a strain rate of a 1mm/min and the geometric measuring of the samples was based on the ASTM E8/E8M [9] procedure are shown on Figure 3 and Table 3.

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 Table 3. Geometric measuring of tensile tests specimen.

Sample	L	В	А	W	С	Т
M350L50F	40mm	16mm	8mm	4mm	10mm	5mm
M350L70F	40mm	16mm	8mm	4mm	10mm	3mm
M350L80F	40mm	16mm	8mm	4mm	10mm	2mm

The extra-reduced specimens were made because the low quantity of available material and a previous work was made with a extra-reduced specimen [10].

## **3 RESULTS AND DISCUSSION**

#### 3.1 Metallographic Analysis

The metallographic analysis of the received samples show a common microstructure, Figure 4, of an high Ni maraging steel, showing no precipitates in the microstructure.



Figure 4. Optical micrograph for the received condition

The optical micrographs for the M350L50F and M350L70F, Figure 5, show that after the post-deformation annealing is observed that the morphology of the grains was not changed, but have a growing on grain size. Due the larger deformation, the recrystalization process could have occurred faster in M350L70F than M350L50F.

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Figure 5. Optical micrographs for the M350L50F and M350L70F conditions

These conditions remain 100% martensitic, independent of applied cold reduction. The grain growing was confirmed by a micro hardness testing were can be observed a softening of the material, the results are shown in Table 4. Carvalho [11] obtained close values to received condition.

		s measuring of the same			
-	Sample	HV1 – 10s ± SD			
-	Received	$340 \pm 8.5$			
-	M350L50F	$325 \pm 6.5$			
	M350L70F	314 ± 8.7			

Table	4.	Micro	hardness	measuring	of the samples	
-						

In the M350L80F condition, the microstructure showed a very small intermetallic precipitation, the electronic micrograph of this condition is shown on Figure 6.



Figure 6. Electron micrograph of the M350L80F condition.

This condition achieved hardness higher than the previous conditions, 400 HV1 – 10s. Previous papers related a intermetallic precipitation on maraging steels after the post-cold rolling annealing [12] identified as Laves phase, but in a cobalt free low Ni maraging steel, according to phase diagram, get by Thermocalc software, Figure 7, is not possible the precipitation of Laves phase in Maraging 350 steel.



Figure 7. Phase diagram to Maraging 350 steel

Due the composition of Maraging 350 steel and the morphology of the precipitate it could be the  $\mu$  phase, related Ni super-alloys [13] and HAZ of duplex steels welding [14]. The micrograph allows seeing a ultra-grains refinement, that could be causes by competition between the precipitation and recrystalization.

## 3.2 Texture and Anisotropy Analysis

The section of  $\varphi_2$ =45° of the ODFs for the annealed samples is shown in Figure 8.



Figure 8.  $\phi_2$ =45° sections of the ODFs for the annealed samples, a)M350L50F, b)M350L70F and c)M350L80F

The Figure 8 show that increasing the pre-annealing deformation there a reduction on cube on edge component and strengthen on the  $\gamma$  fiber. The strength  $\gamma$  fiber is good deep drawing because induces less force to draw and the slight presence of preferential directions is good to avoid the ear formation.

Analyzing the Lankford's coefficient, shown on Table 5, it was found that the planar anisotropy coefficients increase whit the deformation.

 Table 5.
 Lankford's coefficients, planar and normal anisotropy coefficients for the specimens measured by tensile testing.

Sample	R <sub>0</sub>	R <sub>45</sub>	R <sub>90</sub>	R <sub>m</sub>	ΔR
M350L50F	1,03	1,11	0,98	1,0575	-0,105
M350L70F	1,12	1,17	1,2	1,165	-0,01
M350L80F	1,7	1,1	1,22	1,28	0,36



The increasing on Lankford's coefficients is unexpected since the thickness of contributes to resistance to wall thinning in deep drawing, but it could be justified due a better crystallographic texture on most cold rolled samples.

## 4 CONCLUSION

The microstructure of the maraging 350 is strongly influenced by the cold deformation before the annealing, but his hardness keeps close to initial value if there is not precipitation. The annealing texture is strongly influenced too, moving from a predominant cube on edge texture to a sharp  $\gamma$  fiber, leading to good results in Lankford's coefficients. It was found that the cold rolling before the annealing improves the planar and normal anisotropy coefficients, but a larger deformation could lead to a intermetallic precipitation of a probable  $\mu$  phase. So the best condition to make a deep drawing was found as a cold rolling to 70% in reduction of thickness followed by a annealing with a small cooling rate.

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