

Hot Tensile Behavior and Fracture Characteristics of a Plasma Nitrided Maraging 300 Steel

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Abstract. The influence of plasma nitriding of a maraging 300 steel on mechanical properties at high temperature has been studied. Samples were tensile tested at 600°C in four conditions: solution treated (MAR-S), solution treated and aged (MAR-SA), solution treated and plasma nitrided (MAR-SP) and solution treated, aged and plasma nitrided (MAR-SAP). In the same sequence, the yield strength and ultimate tensile strength increased slightly respectively from 1073 to 1189 MPa and 1174 to 1301 MPa, an increase of about 10% due to plasma nitriding. All the samples presented similar values of elongation, around 18%, but the cross section area reduction decreased significantly by plasma nitriding from ~70% for MAR-S and MAR-SA to ~45% for MAR-SP and MAR-SAP, that is an decrease of 36% in average. This decrease is attributed to brittle fracture nucleated at 50 µm thick iron nitride layer. The inner fracture surface of the tensile tested specimens was predominantly ductile presenting characteristic microcavities.

Introduction

The motor case is the main load-bearing structural member of a solid propellant rocket. It functions as a container for the propellant, acts as the combustion chamber during propellant burning, and performs as the main structural member of the flight vehicle. [1]. Maraging are ultra-high resistant steels with Ni-Co-Mo-Ti presenting a broad range of application in key areas such as nuclear and aerospace industries and its development is part of the priority list of advanced materials research in Brazil [2]. Maraging steels are nowadays in study to replace the current 300M steel in use as a motor case of Brazilian satellite launch vehicle. The current manufacturing process of the rocket motor cases produced with 300M steel has shown some issues related to production cost and waste disposal during the heat treatments: stress relief (620 °C/3 h), normalization (540 °C/1 h and 930 °C/0.5 h), quenching (980 °C/1 h) and double tempering (280 °C/2 h) [3]. The components production using maraging steels is quite simple requiring only a solution annealing (820 °C/1 h) followed by an aging (480 °C/3 h) [4]. Besides that, the maraging steel is very easy to mechanically conform (rolling, machining) in a martensitic state and no dimension variation occurs upon aging heat treatment, that is, the technology of “near net shape” production could be used very easily.

Data available on the mechanical properties of maraging steel at elevated temperature are very scarce. Specialized applications of the steel, such as the motor case of a solid propellant rocket,

occasionally demand short-time exposures at high temperatures and it is desirable to have data on the hot tensile behavior of the material during such service conditions [5-8].

Nitriding is a surface treatment process involving the introduction of nitrogen into the surface of steel, which produces a modified layer with excellent properties such as high hardness, good wear and corrosion resistance and strength increase [9, 10]. However, conventional nitriding process is usually carried out at high temperature for a long time, which is beyond the aging temperature and time of maraging steel and would result in the overaging and reversion of martensite to austenite. By the other hand, plasma nitriding could be carried out at the same temperature (or lower) of the aging temperature resulting in simultaneous nitriding plus aging reducing production cost, one of the concern of 300M steel [1, 10, 11]. In addition, overaging and reversion of martensite to austenite could be avoided by this double function process [12].

In the present work, the influence of plasma nitriding of a maraging 300 steel on its tensile properties at 600 °C and fracture surfaces has been investigated.

Experimental Procedure

In this work a maraging 300 steel solution treated at 820 °C during 1 h and then air-cooled was used. Table 1 shows the chemical composition of the steel.

Table 1 – Chemical composition [wt.%] of the maraging 300

Co	Mo	Ni	Ti	Al	C	S	P	Si	Mn	Fe
9.37	4.94	19.00	0.63	0.08	0.008	0.002	0.004	0.06	0.01	Balance

From the solution treated stock specimens were machined to an 18.5 mm gauge length by 3.0 mm in diameter and some of them were submitted to an additional age hardening treatment at 480 °C for 3 h. Some of the samples in both conditions were then plasma nitrided in the furnace under vacuum. Plasma was obtained by passing the gas mixture of H₂ and N₂ in the ratio of 3:1 under vacuum. Plasma nitriding was carried out at 480 °C for 3 h for both conditions. The specimens submitted to the hot tensile tests are Solution Treated (MAR-S), Solution Treated and Aged (MAR-SA), Solution Treated and Plasma Nitrided (MAR-SP) and Solution Treated, Aged and Plasma Nitrided (MAR-SAP). An Instron Model 3383 testing machine coupled with an open vertical cylindrical furnace Model SP-16-2230 was used for the hot tensile tests. Temperature control during the tests was done using two chromel-alumel type thermocouples in contact to reduced section surface of the specimen. The hot tensile tests were carried out at 600 °C in air atmosphere with average nominal strain rate of 0.5 mm/min. Software Blue Hill 2.6 was used for data acquisition. One tensile test was carried out for each of the four conditions.

Microstructural examination was carried out on Carl Zeiss H-PL optical microscope. Cross sections from nitrided and un-nitrided samples were ground, polished (final polishing step: 1 µm diamond suspension) and etched using FeCl₃ 10% (5 g FeCl₃ in 45 mL of water) at room temperature for about 2 s. Examination of the fracture surfaces of the hot tensile tested specimens was done using a VEGA 3-TESCAN scanning electron microscope.

Results

Fig. 1 shows optical micrographs of un-nitrided and plasma nitrided specimens revealing a uniform and continuous case hardened nitrided layer and the typical martensite plate-like microstructure of maraging steels in the un-nitrided core. The thickness of 50 µm was found in the nitrided layer for all nitrided samples. In the light optical micrographs (Fig. 1), no difference in the microstructure of the un-nitrided core of the age-hardened sample can be observed as compared to that of the only solution annealed sample.

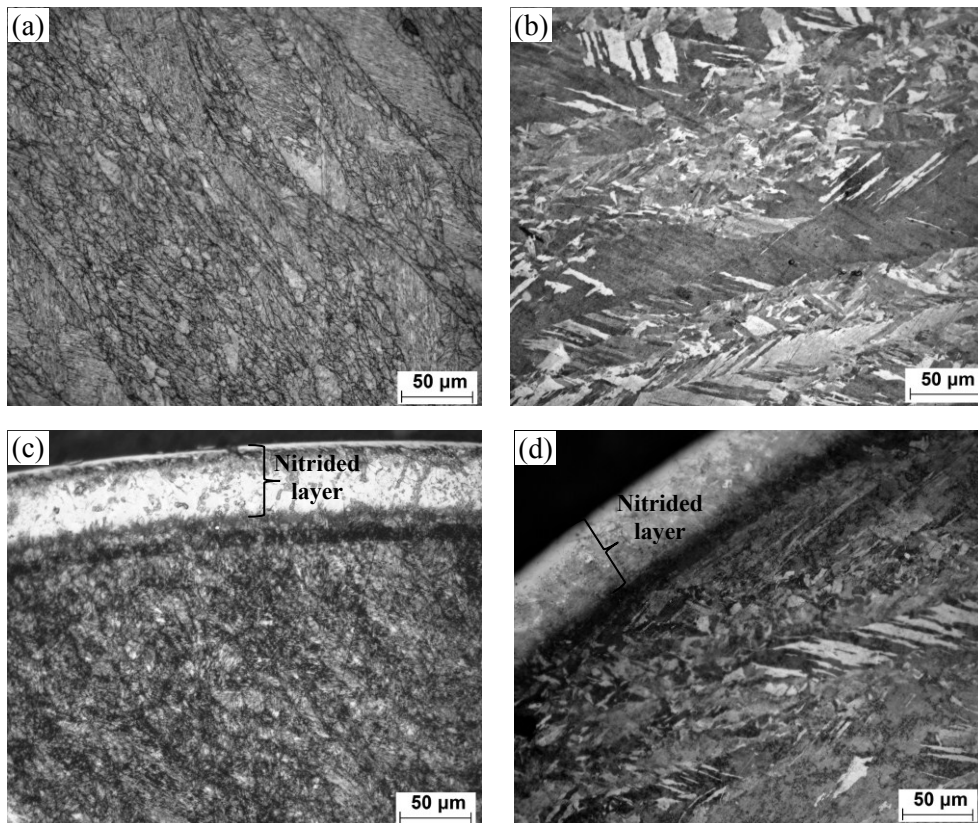


Fig. 1 - Optical micrographs of maraging 300 (a) MAR-S, (b) MAR-SA, (c) MAR-SP and (d) MAR-SAP

Fig. 2 shows the tensile test curves at 600 °C of maraging 300 steel specimens after different thermal and thermochemical treatments. The mechanical properties determined from the tensile test curves is summarized in Table 2.

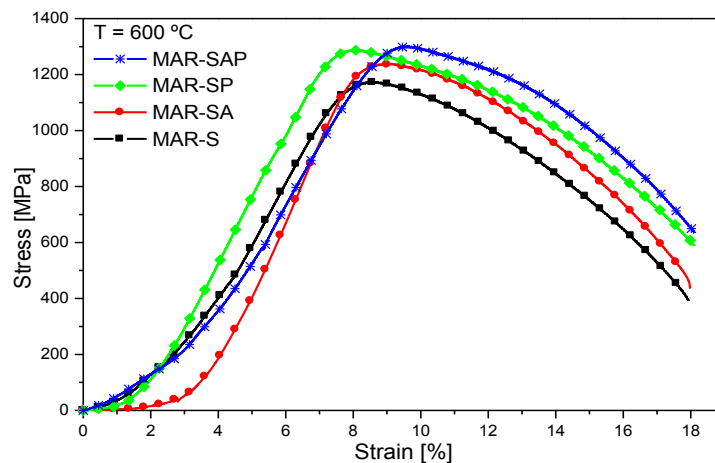


Fig. 2 - Stress-strain curves of maraging 300 obtained from tensile tests at 600 °C: MAR-S, MAR-SA, MAR-SP and MAR-SAP

Table 2 – Yield strength, σ_e , the ultimate tensile strength, σ_{max} , the total elongation, ϵ_{total} , and reduction in area, RA, as determined from the tensile tests carried out at 600 °C with specimens nitrided and un-nitrided of maraging 300 steel

Material	σ_e [MPa]	σ_{max} [MPa]	ϵ_{total} [%]	RA [%]
MAR-S	1073	1174	18.3	69.9
MAR-SA	1110	1239	17.9	70.5
MAR-SP	1163	1289	17.6	47.2
MAR-SAP	1189	1301	17.5	43.2

According to Fig. 2 it can be noticed that the shapes of stress-strain curves are similar, confirmed by the values of σ_e , σ_{max} , and ϵ_{total} shown in Table 2. The occurrence of non-linear stress-strain behavior in the conventional elastic region can be associated with the overaging, since the tests were performed at a temperature close to the austenite formation ($A_s = 623 \text{ }^\circ\text{C}$) [8]. MAR-S has σ_e and σ_{max} at room temperature of 790 and 1010 MPa, respectively [4], and the higher values of σ_e and σ_{max} at 600 °C are due to rapid age hardening at high temperatures, and this result is related to the softening caused by test temperature and hardening caused by precipitation. The yield strength and the ultimate tensile strength showed an increase of 5 to 10% with plasma nitriding when comparing with aging treatment. This result can be associated to the hard layer of iron nitrides in the surface that works as a barrier to the plastic deformation.

From Table 2 it can be noticed also that the ductility (RA) of plasma nitrided samples were reduced in an average of 36% when compared with un-nitrided samples. This reduction in ductility can be also shown by fractography analysis. The fracture surfaces of the maraging 300 steel specimens that were tensile tested at 600 °C are shown in Fig. 3 to 5.

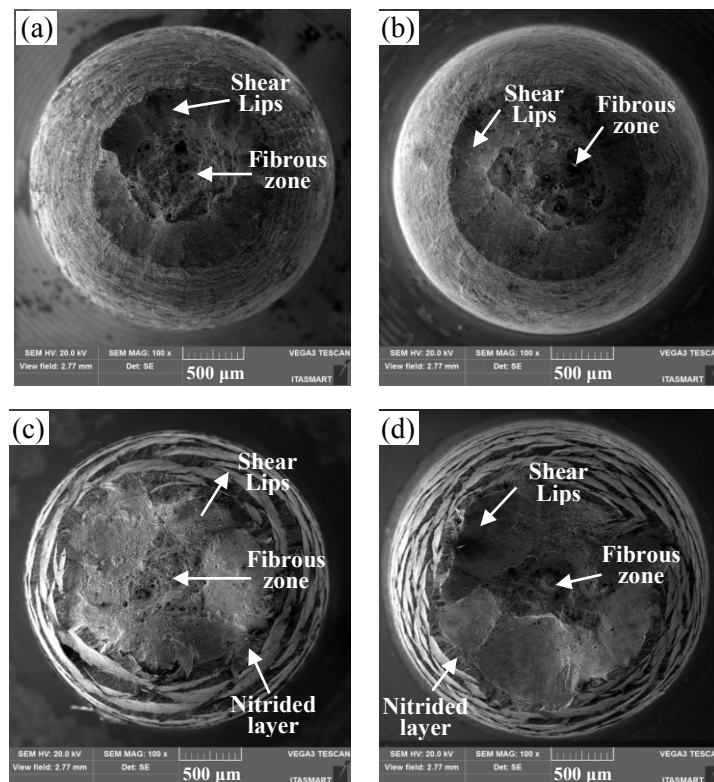


Fig. 3 - Scanning electron micrograph overview of fracture surface after tensile tests at 600 °C of (a) MAR-S, (b) MAR-SA, (c) MAR-SP and (d) MAR-SAP

From Fig. 3 it can be seen that the nitrided and un-nitrided specimens showed a cup-and-cone fracture morphology after hot tensile tests, characteristic of a ductile type of failure, with the general view of the tensile fracture surface consisting of dimpled rupture surrounded by shear lips as indicated by arrows in Fig 3.

In the peripheral regions of the fracture surface (Fig. 4) it can be seen that the nitrided and un-nitrided specimens showed a 45° shear lip with the elongated C-shaped dimples due to tensile shearing near the end of the fracture, opposite to the origin. However, observations from the nitrided samples MAR-SP (4c) and MAR-SAP (4d) revealed the nitrided layer with typical brittle fracture with flat regions and radial cracks.

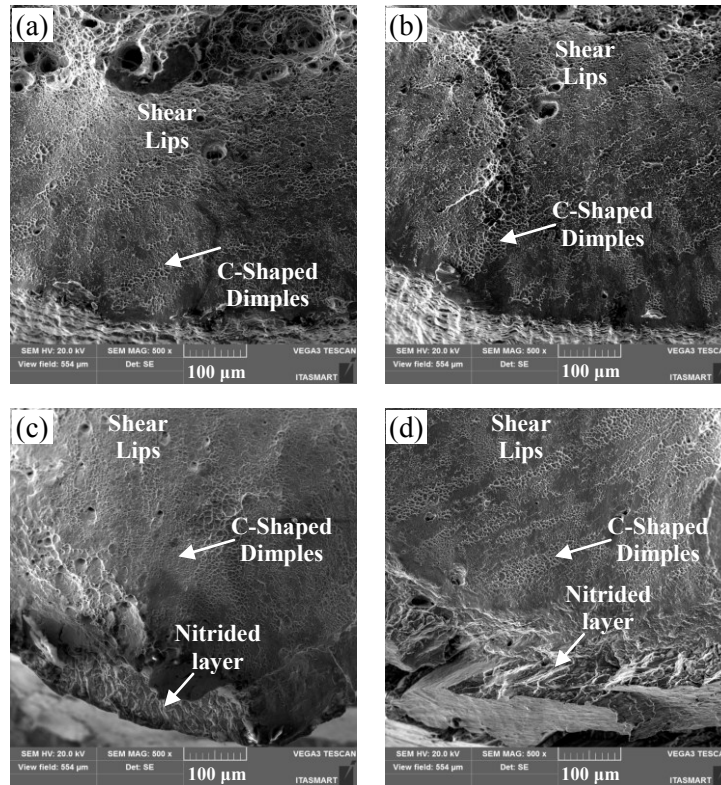


Fig. 4 - Scanning electron micrograph of peripheral region of fracture surface after tensile tests at 600 °C of (a) MAR-S, (b) MAR-SA, (c) MAR-SP and (d) MAR-SAP

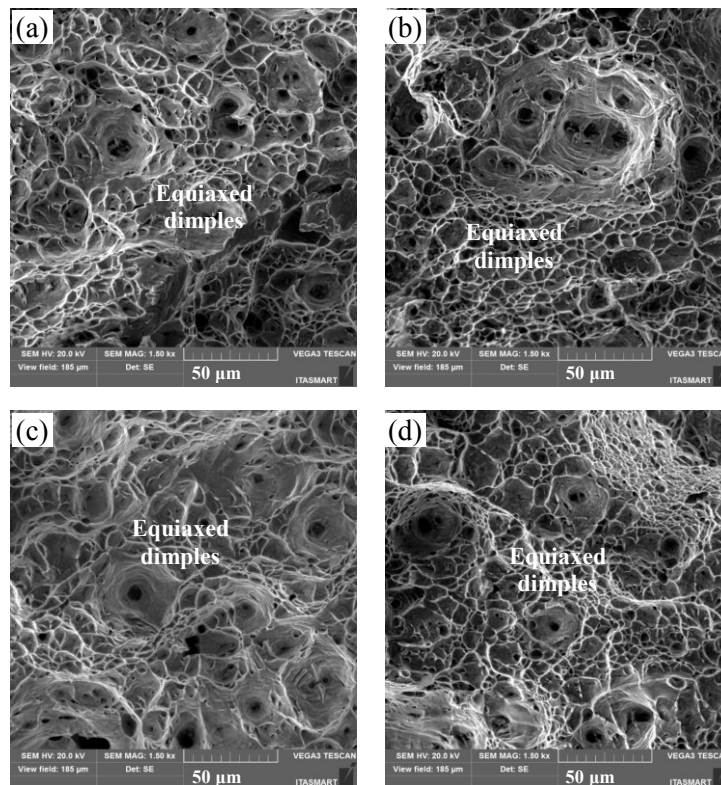


Fig. 5 - Scanning electron micrograph of center region of fracture surface after tensile tests at 600 °C of (a) MAR-S, (b) MAR-SA, (c) MAR-SP and (d) MAR-SAP

The center region of fracture surfaces (Fig. 5) of the nitrided and un-nitrided specimens showed equiaxed dimples with a bi-modal structure.

Conclusion

Based on the present work on maraging 300 steel, typical hot tensile curves were found with similar shapes for nitrided and un-nitrided samples. Nitrided samples showed a higher yield strength and the ultimate tensile strength when compared to un-nitrided samples, and it can be associated to hard surface layer of the compound layer. Dominant type of failure was ductile showing a typical cup-and-cone fracture morphology, consisting of equiaxed and bi-modal dimples in the fibrous zone surrounded by 45° shear lip for nitrided and un-nitrided samples. However, the plasma nitriding treatment decreased the ductility (reduction in area) by 36% as a result of premature cracking of the compound layer.

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