High Temperature Mechanical Behavior Of Plasma-Nitrided Inconel 625 Superalloy

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Abstract: INCONEL 625 nickel-based superalloys present limitations for use at temperatures above 600°C. For this reason, protective coatings can be used as barriers to avoid both nucleation and crack propagation. The aim of this work is to evaluate the mechanical properties at high temperatures of non-nitrided and plasma-nitrided samples of the INCONEL 625 superalloy. The microstructural characterization of the nitrided layer was performed with the aid of optical microscopy (OM) and X-ray diffraction (XRD). Tensile tests were performed between 600 and 1000°C and deformation rates between 0.0002 and 0.002 s^{-1}. The results have shown that nitrided sample present expanded fcc phase and chromium nitride (CrN) phases. Tensile tests showed that there was no significant difference in the yield strength and elongation between non-nitrided and plasma-nitrided samples at the same temperatures. Serrated stress–strain behavior was observed in the curves obtained at 600 and 700°C, which was associated with the dynamic strain aging effect. At 600°C, the increase in strain rate promoted an increase of the amplitude and oscillation frequency of the stress.

Keywords: Inconel 625, plasma-nitriding, superalloys, mechanical properties

1. Introduction

Nickel-based superalloys generally present high mechanical properties, good creep, fatigue and corrosion resistance and are able to work at high temperatures. These properties are reached by the combination of a solid solution austenitic matrix (γ phase) with high volumetric fraction of coherent precipitates such as $Ni(Ti, Nb,Ta)_3Al$, which is commonly known by gamma-prime phase. These materials are also present at up to 75% weight percent of Ni and up to 30 weight percent of Cr. [1]

Inconel 625 is a nickel-chromium-molybdenum alloy with excellent oxidation resistance, outstanding strength and toughness from cryogenic to high temperatures. Alloy 625 also has exceptional fatigue resistance. The high amount of Cr (21 wt. %), Mo (9 wt %) and Nb (4 wt %) increases the resistance of γ phase without the necessity of solution and aging heat-treatments [2]. It also promotes an increasing of up to 40% for both yield and ultimate tensile strength at room temperature when compared with the 600 alloy, for the same elongation. At high temperatures (870^{o}C for example), the increasing of the mechanical properties of the alloy 625 is between 50 and 120% higher than the alloy 600 [2,3]. However, the γ phase presents MC and M_6C carbides with high amounts of niobium and molybdenum.

This alloy also resists a wide range of severely corrosive environments and is especially resistant to pitting and crevice corrosion. Due to the outstanding corrosion resistance, this precipitation hardenable alloy is commonly used in chemical processing, aerospace and marine engineering, pollution-control equipments, and nuclear reactor applications.

Despite the notable progress in the development of the mechanical resistance at high temperatures of the Inconel alloys, limitations can occur when these alloys are used in temperatures higher than 600°C. The Inconel 625 superalloy presents better weldability than many others nickel-base alloys. In spite of their otherwise excellent properties, Inconel 625 has poor wear characteristics. Thus, surface treatments can be used in order to improve the tribological behavior without affecting corrosion resistance [4].

Plasma nitriding has been traditionally used as a surface treatment for several nickel-based alloys to create protective coatings that can be used as barriers for both nucleation and crack propagation when these materials are applied for long time at high temperatures. However, this process is normally conducted at relatively high temperatures resulting in tempering, affecting temperature-sensitive microstructures and/or producing non-desirable equilibrium phases [4].

For example, these nitriding treatments of the Cr-containing alloy, such as the Inconel 625 which above 500°C modifies the surface by the formation of chromium nitrides (CrN) and expanded austenitic phase, which degrades the corrosion resistance. Additionally, the main difficulty is the adherence problem that can occur due to the diffusion of the elements that form the coating during the thermal cycles. Based on this, the aim of this work is to study the mechanical properties (through tensile tests between 600 and 1000°C) of plasma-nitrided Inconel 625 superalloy.

2. Experimental Procedure

Bars of the Inconel 625 superalloy (12 mm diameter) were rolled at high-temperatures at the Multialloy Company (São Paulo, Brazil). The nitriding process was performed at 520°C for 12 hours. The material was prepared following conventional metallographic techniques (mounting at 21MPa and 150°C, grinding with SiC sand paper, polishing with diamond paste and finally etching with 100ml HCl + 3ml HNO_3 + 100ml ethylic alcohol for 5 minutes). The microstructure and depth of the samples was analysed with the aid of an optical microscopy Olympus BX51M with Olympus UC30 camera.

Tensile tests were performed in a universal machine (Instron 4400) at temperatures between 600 and 1000°C and deformation rate between 0.0002 and 0.002 s^{-1}. The length and diameter of the specimen were 50 and 5mm, respectively. X-ray diffraction (XRD) experiments were performed using a Rigaku Multiflex difractometer with Cu-Kα radiation, over the 2θ range 20° to 90° at a scanning rate of 1° per minute. Thermodynamic simulations of the phases at each temperature of the mechanical tests were performed with the aid of the JMatPro software (Ni database).

3. Results and Discussion

The result of the chemical analysis of the main elements is as follows: (wt %): 21.5 Cr; 9 Mo; 3.6 Nb; 2.5 Fe; 0.25 Mn; 0.2 Ti; 0.2 Al; 0.05 C; Ni balance. This result is in agreement with ASTM B443-00(2009) [5,6].

Table 1 shows the percentage of the phases obtained by thermodynamic simulations with the JMatPro software. It can be seen that there is a high amount of γ phase at high temperatures and also the presence of 3 types of carbides: MC, M_6C and $M_{23}C_6$. The MC carbides are stable at high temperatures. During aging heat-treatments or service conditions, these particles can decompose slowly, generating carbon that can diffuse into the matrix and initiates important reactions such as MC + $\gamma \rightarrow M_6C/M_{23}C_6 + \gamma'$ in order to form M_6C and $M_{23}C_6$ carbides rich in Cr, Mo and W, which are stable at lower temperatures than MC carbides. The simulation also shows that M_6C carbides are stable at higher temperatures when compared with $M_{23}C_6$ carbides.

Figure 1 shows the micrograph of the nitrided layer for the Inconel 625 superalloy. The depth of the layer is slightly above 4 μm. Figure 2 shows the x-ray diffraction spectra of the nitride sample and the pattern for γ (red) and CrN (green) phases. Figures 3(a), 3(b) and 3(c) show the zoom of positions 1, 2 and 3, respectively. It was noted that nitrided samples presented mainly two phases: γ fcc expanded phase (Fig. 3 – region A) and chromium nitride CrN (Fig. 3 – region B), which were evidenced by peaks at 37.5, 42.5, 63.3 and 76.2°. Additionally, the plasma nitriding of Inconel 625 shifts the (111) peak of the γ fcc phase from 43.6° to 43° and the (200) peak from 50.7° to 49.5°. The different amount of shifting in peak position of (111) and (200) was attributed to the lower atomic density of the latter plane, resulting in larger lattice expansions. The expansion of the lattice in different crystallographic directions depends on the tension level and the elastic constant of the diffraction plane [4].

Figures 4 and 5 show the results of the tensile tests (deformation rate = 0.0002 s^{-1}) between 600 and 1000°C for non-nitrided and nitrided samples of the Inconel 625 superalloy, respectively. The values of yield strength, ultimate tensile strength and elongation are shown in Table 2. As expected, the yield and ultimate tensile strength decreases as the temperature of the test increases. On the contrary, the elongation increases with the increasing of the temperature. This behavior is similar for nitrided and non-nitrided alloys.

Figure 6 to 10 show, in detail, the curves for non-nitrided and nitrided samples of the Inconel 625 superalloy at each temperature (600, 700, 800, 900 and 1000°C). At 600°C, it was not possible to verify significant differences between the curves of nitrided and non-nitrited samples. At 700°C, the ultimate tensile strength of non-nitrided samples was 722 MPa whilst for nitrided sample this value decreased to 613 MPa. The ultimate tensile strength values tend to be higher for the non-nitrided samples at temperatures higher than 700°C. However, the nitrided samples tend to keep high strength as the strain progress, as can be seen in Fig. 9 (at 900°C). This tendency will be investigated in more detail with the creep tests in the future.

Figures 11, 12 and 13 show the results of the tensile tests for the non-nitrided Inconel 625 superalloy at 600°C and different deformation rates: 0.0002 s^{-1} (Fig. 11); 0.001 s^{-1} (Fig. 12); 0.002 s^{-1} (Fig. 13). The mechanical properties at different deformation rates didn't present significant differences. It can also can be seen as a serrated stress–strain behavior which was associated with a dynamic strain aging effect. The increasing in the strain rate promoted an increasing in the amplitude and oscillation frequency of the stress. Different mechanisms can originate this oscillation process, such as dynamic aging and recrystallization, the last one more common at high temperatures [7,8]. At low temperatures, such as 600°C, the main mechanism responsible for the oscillation frequency of the stress was attributed to the Portevin-Le Chatlierou effect [9]. However, it is difficult to determine the mechanisms responsible for this serrated behavior based only on the shape of the stress-strain curves.

4. Conclusions

The results have shown that the nitrided sample presented an expanded fcc and chromium nitride (CrN) phases. The presence of CrN was evident by the two peaks at 37.5° and 42.5°. It can be seen that plasma nitriding of Inconel 625 shifts the (111) peak from 43.6° to 43° and the (200) peak from 50.7° to 49.5°. The different amounts of shifting in peak position of (111) and (200) was attributed to the lower atomic density of the latter plane, resulting in larger lattice expansion. Tensile tests showed that there was no significant difference in the yield strength and elongation between non-nitrided and plasma-nitrided samples at the same temperature. Serrated stress–strain behavior was observed in the curves obtained at 600 and 700°C, which was associated with the dynamic strain aging effect. At 600°C, the increasing in the strain rate promoted an increase in the amplitude and oscillation frequency of the stress.

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Figure 1

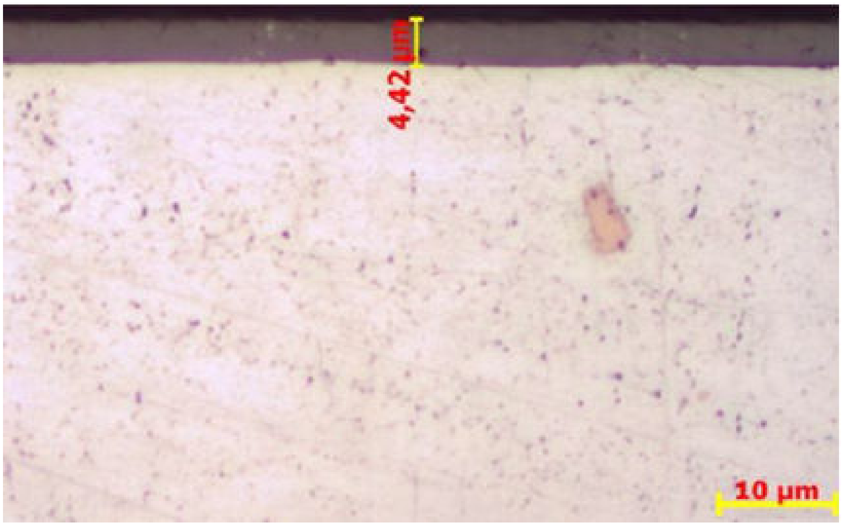
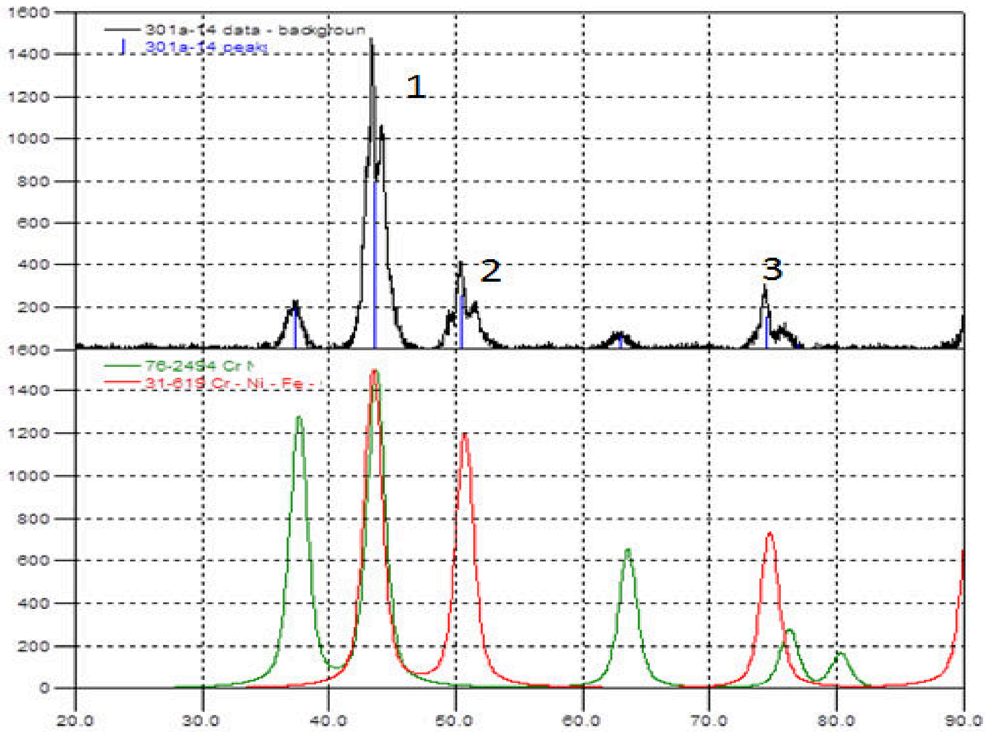


Figure 2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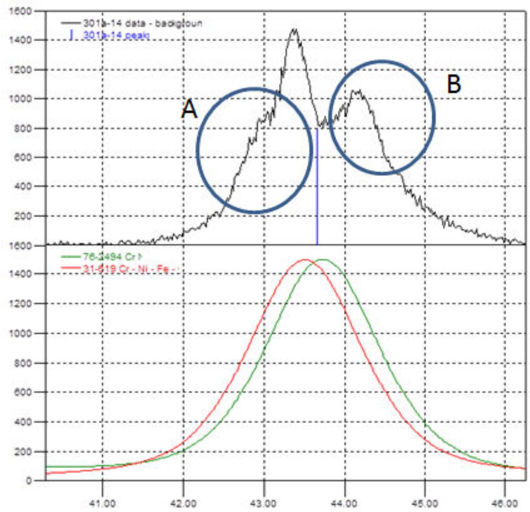
301a-14 peaks
1
2
3
76-2494 Cr N
31-619 Cr - Ni - Fe -
1600
1400
1200
1000
800
600
400
200
0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0

Figure 3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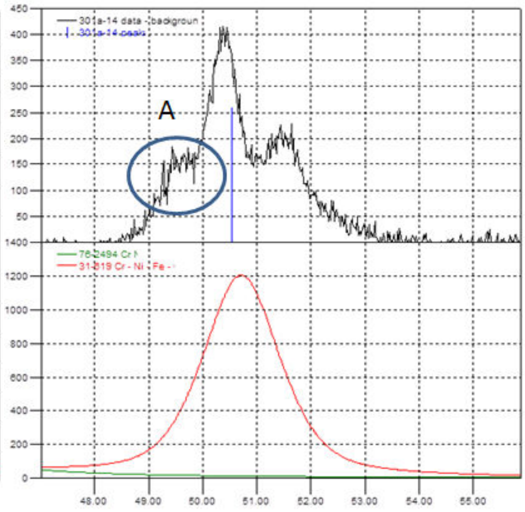
301a-14 peak
A
B
76-2494 Cr N
31-619 Cr - Ni - Fe -

(a)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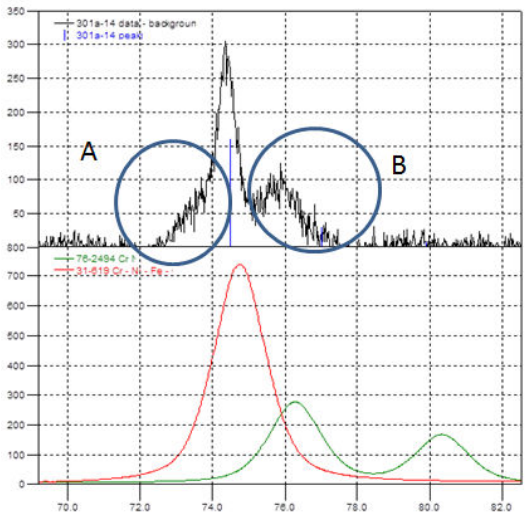
301a-14 peak
A
76-2494 Cr N
31-619 Cr - Ni - Fe -

(b)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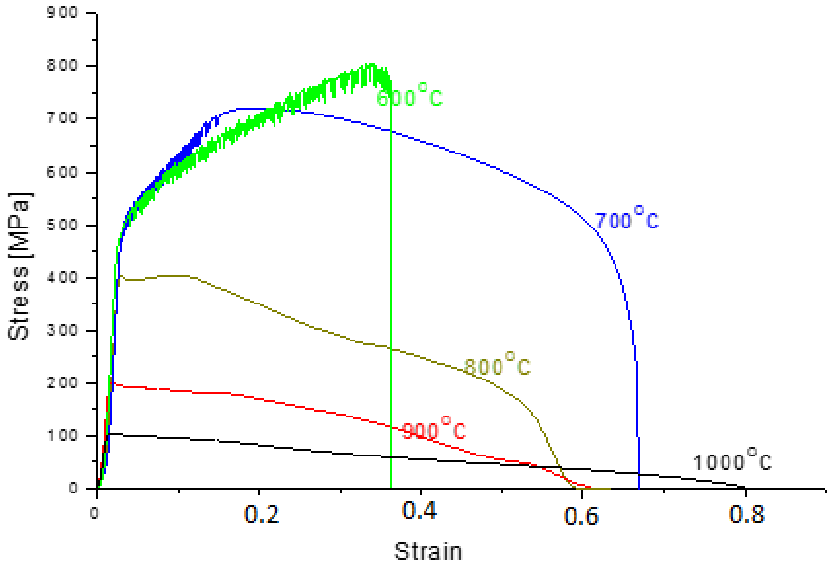
301a-14 peak
A
B
76-2494 Cr N
31-619 Cr - Ni - Fe -

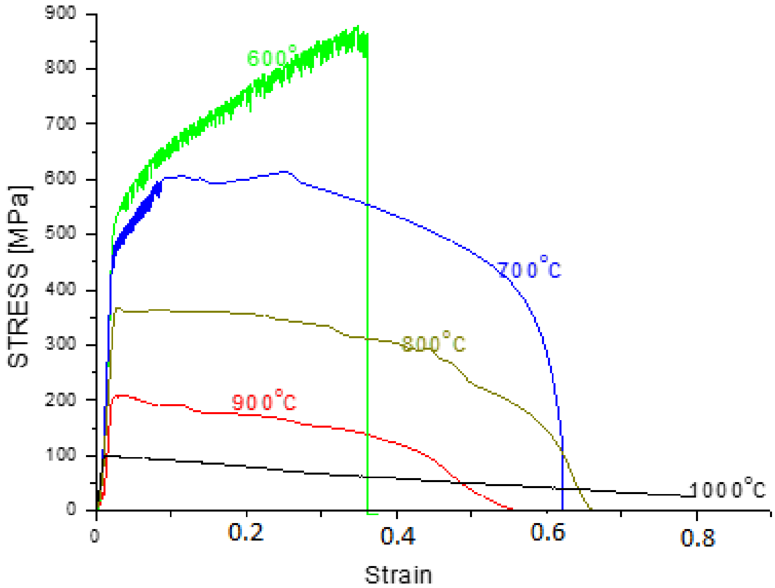
(c)

Figure 4



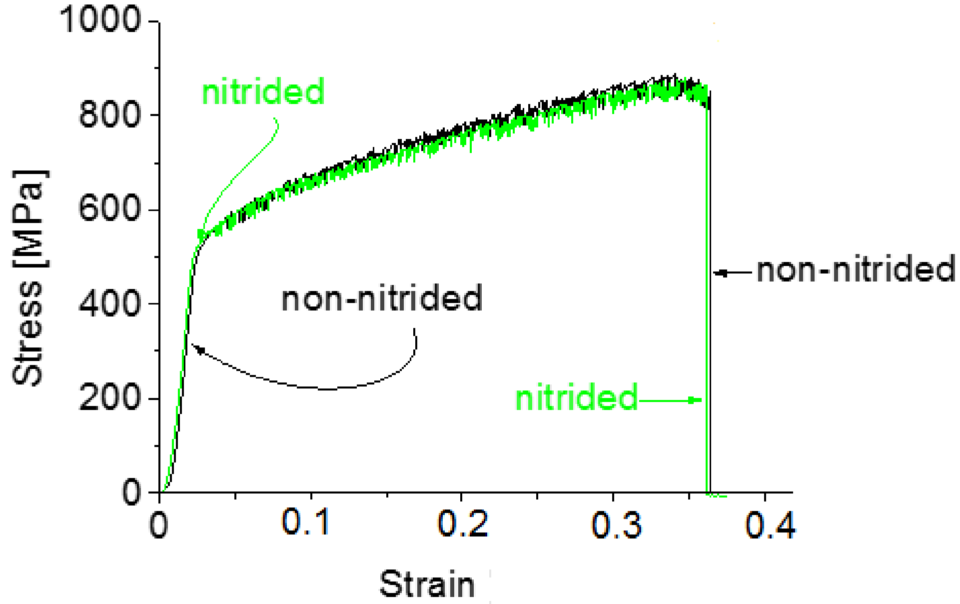
900
800
700
600
500
400
300
200
100
0
Stress [MPa]
0
0.2
0.4
0.6
0.8
Strain
600°C
700°C
800°C
900°C
1000°C

Figure 5



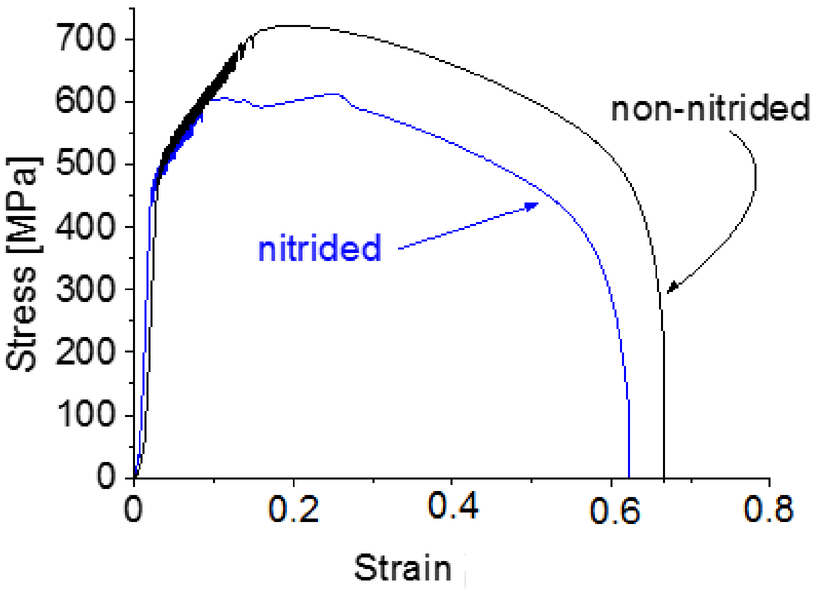
STRESS [MPa]
900
800
700
600
500
400
300
200
100
0
600°
700°C
800°C
900°C
1000°C
0
0.2
0.4
0.6
0.8
Strain

Figure 6



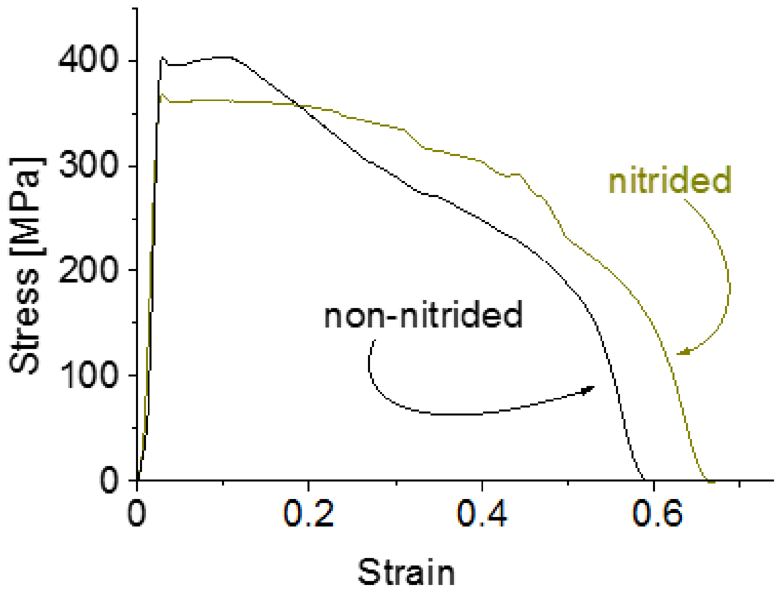
1000
800
600
400
200
0
Stress [MPa]
nitrided
non-nitrided
non-nitrided
nitrided
0
0.1
0.2
0.3
0.4
Strain

Figure 7



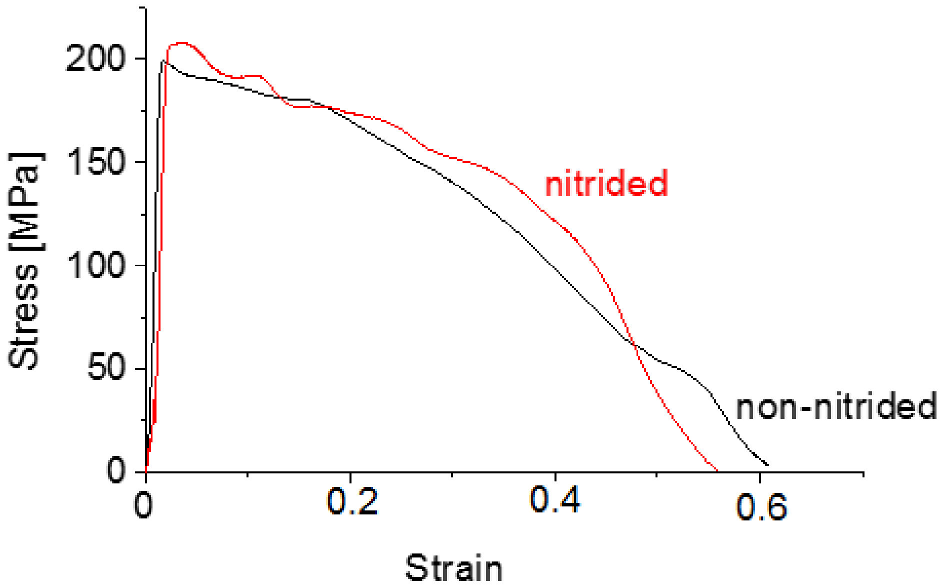
700
600
500
400
300
200
100
0
Stress [MPa]
0
0.2
0.4
0.6
0.8
Strain
non-nitrided
nitrided

Figure 8



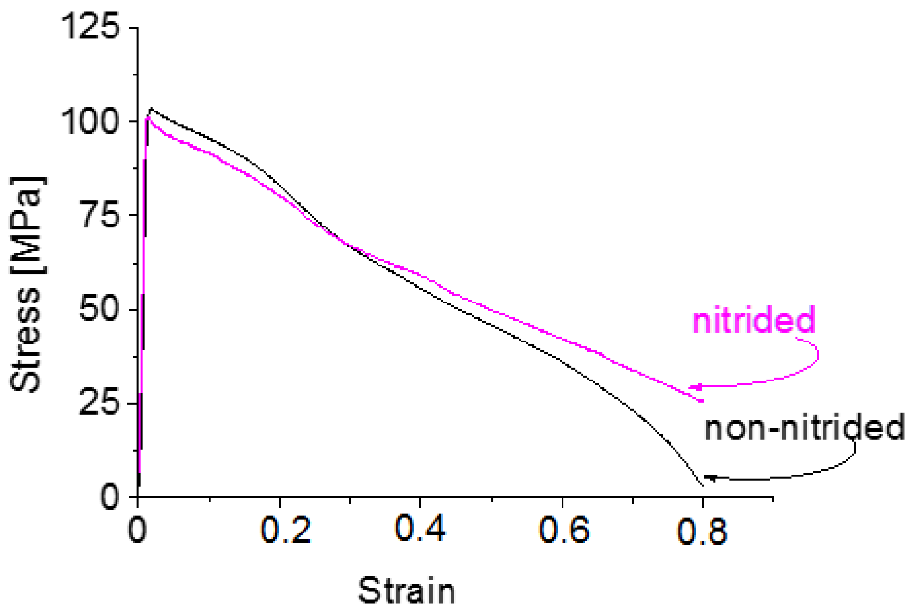
400
300
200
100
0
Stress [MPa]
0
0.2
0.4
0.6
Strain
nitrided
non-nitrided

Figure 9



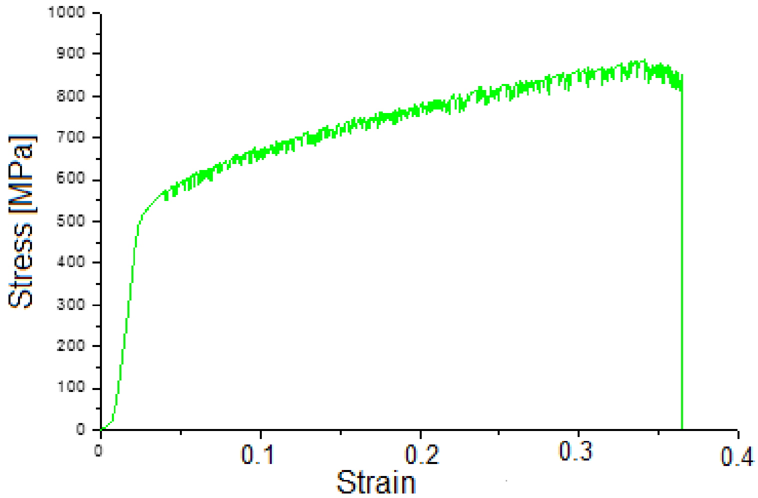
200
150
100
50
0
Stress [MPa]
nitrided
non-nitrided
0
0.2
0.4
0.6
Strain

Figure 10



125
100
75
50
25
0
Stress [MPa]
0
0.2
0.4
0.6
0.8
Strain
nitrided
non-nitrided

Figure 11



1000
900
800
700
600
500
400
300
200
100
0
Stress [MPa]
0
0.1
0.2
0.3
0.4
Strain

Figure 12

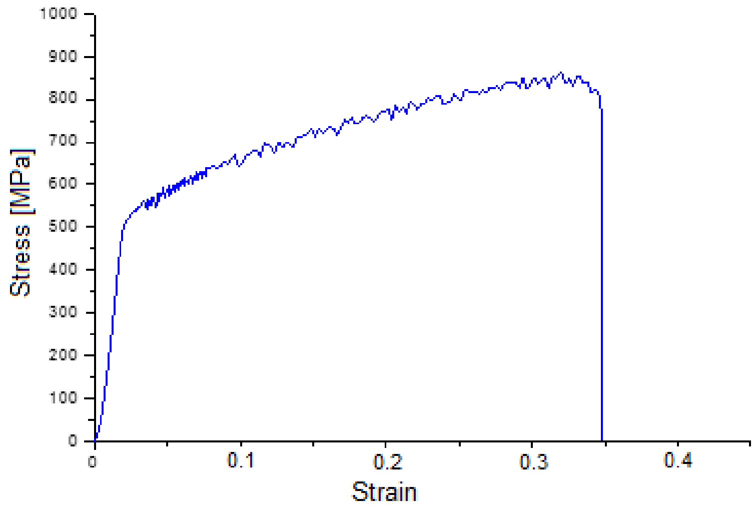
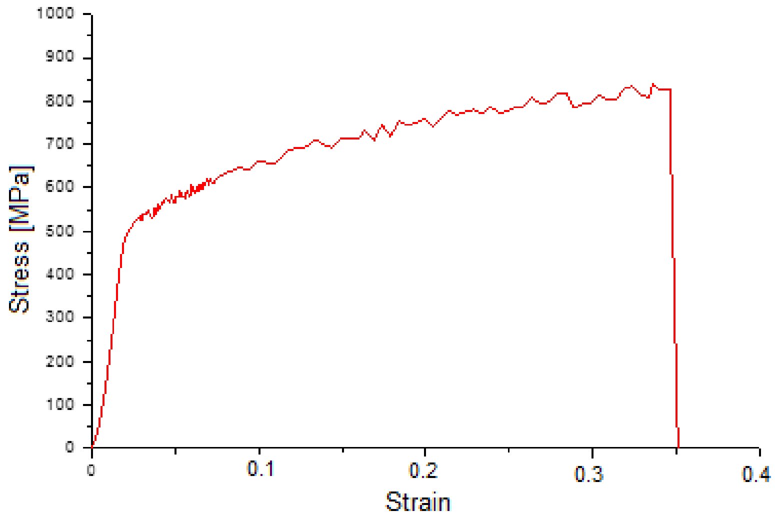

Stress [MPa]
1000
900
800
700
600
500
400
300
200
100
0
0
0.1
0.2
0.3
0.4
Strain

Figure 13



1000
900
800
700
600
500
400
300
200
100
0
Stress [MPa]
0
0.1
0.2
0.3
0.4
Strain

Table 1

T (°C)	Phase fraction (wt. %)						
	γ	μ	δ	γ'	$M_{23}C_6$	M_6C	MC
600	77.11	11.18	9.32	1.41	0.97		
700	83.78	6.99	8.25		0.97		
800	92.22	0.91	5.51		0.52	0.85	
900	97.70		0.52			1.78	
1000	99.10					0.64	0.26

Table 2

Tensile temperature test (°C)	Condition	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)
600	Non-Nitrided	497	887	18
	Nitrided	495	879	19
700	Non-Nitrided	512	722	33
	Nitrided	475	613	32
800	Non-Nitrided	417	417	32
	Nitrided	357	357	34
900	Non-Nitrided	199	199	30
	Nitrided	208	208	28
1000	Non-Nitrided	103	103	40
	Nitrided	101	101	40