Aging characterization of metals for exhaust systems

AGING CHARACTERIZATION OF METALS FOR EXHAUST SYSTEMS

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ABSTRACT−The mechanical characteristics of four materials used in automotive exhaust systems have been compared after an aging treatment to evaluate the combined effects of thermo-mechanical fatigue and corrosion. For this purpose, an experimental aging procedure has been developed. This procedure is composed of chemical, thermal and mechanical cycles, which are combined and repeated to simulate the actual operating conditions of automotive exhaust systems. Three austenitic steels (AISI 309, AISI 316Ti, and AISI 321) and a nickel-based alloy (Inconel 625) are tested. The results show that Inconel 625 and AISI 309 are less affected by the aging process than the other materials.

1. INTRODUCTION

In automotive applications, such as exhaust systems, materials are subjected to harsh operating conditions, such as vibrations, high temperatures, corrosive environments caused by hot exhaust and external conditions (for example, the winter salt deposed on the roads), and mechanical fatigue (Crum *et al*., 1999).

The materials used for automotive exhaust applications are stainless steels and, more recently, nickel alloys (Curà *et al*., 2008).

A common problem arising in exhaust systems is the aging effect, which causes variations in the mechanical properties of these metals.

Several studies in the literature have conducted aging tests on stainless steels and nickel alloys; these studies all simulate specific operating environments.

Crum *et al*. (1999) studied the effects of hot salt attack, stress corrosion cracking and high-temperature embrittlement on materials used for flexible automotive exhaust components; the aging procedure consisted of thermal and chemical attack without the application of mechanical stress.

Choi *et al*. (2008) evaluated the corrosion damage of coated steels in automotive chassis parts over a five-year period.

Pan *et al*. (2001) studied the effects of strain rate and aging on the mechanical properties of sheet steels.

Sahlaoui *et al*. (2004) investigated the behavior of AISI 316L during aging tests lasting up to 80000 h, at temperatures between 550 and 650ºC; in this temperature range, Cr carbides and intermetallic phases are present. The chromium carbide causes the dechromization phenomenon, which is mainly responsible for the intergranular corrosion sensitizing of these steels.

Guan *et al*. (2005) studied the effect of aging at 700ºC on both AISI 321 and AISI 347 austenitic stainless steel welds; the results showed that ductility and toughness may change drastically after long aging times.

Padilha *et al*. (2005) studied the microstructures and mechanical properties of Fe15Cr15Ni austenitic stainless steels. The microstructures and the mechanical properties of cast, hot forged, solution-annealed and aged samples are evaluated and compared. The mechanical properties of the hot forged, solution-annealed and aged (at 700ºC) specimens showed improvements in yield strength, tensile strength and hardness but significant deterioration in ductility (total elongation).

Llanes *et al*. (1996) investigated aging effects on the cyclic deformation mechanisms of a duplex stainless steel, AISI 329. The findings showed that aging of duplex stainless steels at 475ºC induces several microstructural modifications to the mechanical properties, including significant increases in the yield strength and ultimate tensile strength and decreases in the fracture ductility.

Shanker *et al*. (2001) investigated the microstructural and mechanical properties of the Inconel 625 superalloy after 60000 h of service at approximately 873 K and after various durations of exposure to temperatures higher than service temperatures. This paper correlates the mechanical behavior with the microstructural changes occurring as a result of various heat treatments. A decrease in the yielding stress value of the service-exposed material was observed after aging for 1 hour at 1123 K. Aging of re-solution-annealed alloy at 923 K caused an improvement in the

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yielding stress and a reduction in ductility with increasing aging time.

Tamura *et al*. (2000) investigated the mechanical properties of 8Cr-2WVTa steel aged for 30000 h at 400-650ºC. The findings showed that both the Vickers hardness number and the creep strength decrease with aging at 650ºC and that the ductile-brittle transition temperature increases with both aging time and aging temperature.

Girone *et al*. (2005) investigated the effect of artificial seawater on the low-cycle fatigue behavior of superduplex stainless steels in air and in artificial seawater. The results showed a remarkable reduction in fatigue life in the presence of the aqueous solutions, especially for high-amplitude strains.

More recently, Bei *et al*. (2010), Zhang *et al*. (2009) and Kuo *et al*. (2009) conducted aging tests on alumina-forming austenitic stainless steels, 2101 stainless steel and the nickel alloy Inconel 718, respectively.

The studies referenced above involve experiments related to the mechanical characterization of metals; however, to the best of the authors' knowledge, to date no studies have been conducted on the combined aging effects found in exhaust systems.

The aim of this paper is to evaluate the effects of thermomechanical fatigue and corrosion on mechanical properties of materials used in exhaust systems in order to provide a quick and practical comparison of the performances of these materials. For this purpose, an aging procedure has been developed. The aging procedure presented here is divided into five steps to simulate the mechanical and thermal stresses and the chemical attacks to which the flexible joints are subjected during their lifetimes.

This procedure has been performed on four different materials: three austenitic stainless steels and one nickel alloy. The comparison between these materials, in terms of the variations in both the mechanical properties and the effects of corrosion, has been performed using the CTQs approach (Zonfrillo *et al*., 2008).

2. MATERIALS AND METHODS

In the present work, three stainless steels (AISI 309, AISI 316Ti, and AISI 321) and a Ni superalloy (Inconel 625) have been characterized. Table 1 shows the chemical composition of these materials.

Stainless steel AISI 309 offers high mechanical resistance at high temperature and an enhanced corrosion resistance because of its high percentages of Ni and Cr. This alloy is used for furnace parts, high temperature containers, welding wires, and firebox sheets (Di Caprio, 1977).

AISI 316Ti is obtained by adding titanium to AISI 316 in order to enhance its heat resistance, and thus, AISI 316Ti is used in flexible chimney liners. The addition of Mo to the alloy prevents specific forms of corrosion, such as pitting and stress corrosion. AISI 316Ti offers high mechanical resistance and high temperature creep resistance (Di Caprio, 1977).

AISI 321 is a chromium-nickel stainless steel that offers improved intergranular-corrosion resistance. This steel is resistant to atmospheric corrosion, sterilizing solutions, many organic chemicals, and a wide variety of inorganic chemicals. The 321 type is suited for applications with temperatures between 440ºC and 900ºC, such as aircraft collector rings, exhaust manifolds, expansion joints and high temperature chemical process equipment (American Society for Metals, 1986).

Inconel 625 is a nonmagnetic, corrosion- and oxidation-resistant, nickel-based alloy. This material offers high strength and toughness in the temperature range of 196ºC to 1100ºC. These properties result from the solid solution effects of the refractory metals columbium and Mo in a Ni-Cr matrix. Alloy 625 has excellent fatigue strength and stress-corrosion resistance and is especially resistant to pitting and crevice corrosion and cracking due to chloride ions. Typical applications for alloy 625 have included heat shields, furnace hardware, gas turbine engine ducting, combustion liners and spray bars, chemical plant hardware and special seawater applications (Special Metals, 2004).

The three stainless steels studied are normally used as materials in the production of exhaust systems; in this study, the Inconel 625 alloy is tested and compared with the stainless steels to evaluate the potential for the use of this material in the production of exhaust systems.

Specimens have been obtained from 0.2 mm-thick sheets after a mechanical treatment of pickling and a thermal treatment of full annealing.

The aging procedure has been designed to simulate the actual operating conditions of exhaust systems and to compare the performances of the four materials.

The specimens were obtained by dinking from 0.2 mm-thick sheets of each selected material; for each material, 20 specimens were collected (80 specimens in total). The geometry of the specimens is in accord with DIN/EN

Table 1. Chemical composition of the studied materials.

Alloy	Fe	Si	Ni	Cr	Ti	Mo	Others
AISI 309	Bal	1.87	11.09	19.16	-		Mn – 0.93
AISI 321	Bal	0.50	10.20	18.14	0.40	-	-
AISI 316Ti	Bal	0.62	11.14	17.34	0.35	2.08	-
INCONEL 625	3.08	0.08	Bal	21.98	0.25	9.19	Nb – 3.52

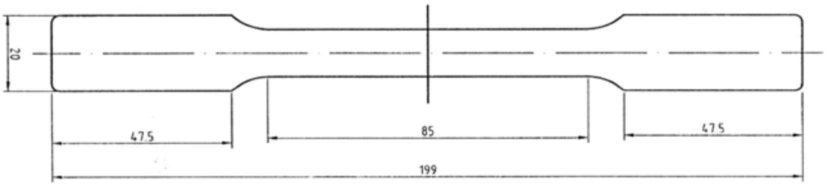

Figure 1. Geometry and dimensions of the specimen according to DIN/EN 10002 (DIN 50114/50125).

10002 standard (Figure 1).

The first aim of the testing procedure is to evaluate and compare both the mechanical resistance and the ductility of the materials as a function of the aging level.

The second aim is to evaluate the corresponding corrosion resistance. The aging test has been designed to simulate the operating conditions (in terms of mechanical fatigue, thermal fatigue and corrosion) of the flexible components in exhaust systems.

The basic mechanical properties of the materials have been determined by tensile testing five specimens of each material at room temperature and 500ºC (monotonic tensile test).

The following parameters are obtained according to DIN/EN 10002: the ultimate tensile strength (R_m), the 0.2% offset yield strength (R_{p02}), the ultimate elongation (A) and the variation of the specimen width (neck) (Z) from the initial state to fracture. In the present work, Z has been defined by considering only the variation of the width, instead of the variation of the specimen section (as described in the DIN/EN 10002 standard), because the variation in the thickness of the specimen is negligible with respect to the variation in width. Z may be expressed as $Z = ((S_0\text{-}Su)/S_0)*100$, where S_0 is the initial width of the calibrated section of the specimen and Su is the minimum width after failure.

The first three parameters (R_m, R_{p02} and A) have been used to measure the variation in the mechanical properties, and the fourth parameter (Z) has been used to evaluate the variation in ductility.

The aging test consists of five steps that are repeated as many times as desired. In the present case, the five steps have been repeated 20 times.

First step: The specimens are subjected to 5 cycles of tensile stress at 500ºC at a loading level corresponding to 80% of R_{p02} and then back to the unloaded condition.

Second step: The specimens are placed in a furnace at 500ºC for two hours and then cooled at room temperature.

Third step: Same as the first step.

Fourth step: The specimens are removed from the furnace used to heat the specimens to a temperature of 500ºC and then sprayed with a saline solution. The solution is composed of NaCl, MgCl and CaCl2 (30% solution in water: 40 parts by weight of NaCl, 1 part by weight of CaCl2, and 1 part by weight of MgCl2) to simulate the chemical mixture used on roads to prevent the formation of ice.

Fifth step: The specimens are placed in a saline cell for 18 hours.

To determine the effect of the degree of aging on the mechanical resistance, in every third procedure (*First step through fifth step*), three specimens of each material are tensile tested at room temperature.

Tensile tests were performed using the MTS Qtest 10 elite machine (10 kN load cell). A saline cell (SECI), a SAPIM electric furnace (up to 1000ºC) and a Watlow heater have been used in these experiments.

The results have been analyzed from both mechanical and corrosion points of view by using the CTQs approach (Zonfrillo *et al*., 2008) to compare the performance of the materials; this approach consists of calculating an index value for each characteristic. In this paper, five characteristics have been considered, and the corresponding indices have been calculated as follows:

- tensile strength index $I_{Rm} = R_{m\_a} - R_{m\_i}$;
- 0.2% offset yield strength index $I_{Rp02} = R_{p02\_a} - R_{p02\_i}$;
- ultimate elongation index $I_{Rp02} = R_{p02\_a} - R_{p02\_i}$;
- variation of the specimen width index $I_A = A_{\_a} - A_{\_i}$;
- corrosion index I_c.

The subscript *a* represents the value obtained from the non-aged material at room temperature, and the subscript *i* represents the value obtained at the end of the aging test for the i^{th} material.

The corrosion index has been obtained by considering four types of corrosion phenomena: general corrosion, intergranular corrosion, pitting corrosion and hot salt corrosion (Speranza, 2006). For each corrosion phenomenon, two penalties have been assigned: one penalty representing

Table 2. Penalty assigned to the corrosion phenomena.

Danger	**Penalty**
Not dangerous	1
Dangerous	2
Very dangerous	3
Entity of the corrosion	**Penalty**
Not present	0
Small traces	0.5
In much of the area	1
Large areas	1.5
Widespread	2

the level of danger related to the corrosion, and the other representing the corrosive entity, according to Table 2 (Speranza, 2006).

The ranking of the analyzed materials is obtained by using the target function OF. The OF function is calculated by summing the products of the normalized values of the indices $CTQs_i$ and the associated weights C_i:

$$OF = \sum_i C_i \cdot CTQs_i$$

According to Speranza, 2006, the weights used in this work are as follows: $C_{Rp02} = 2$; $C_{Rm} = 2$; $C_A = 3$; $C_Z = 1$ and $C_{corrosion} = 3$.

3. RESULTS

The results of the preliminary static characterization are shown in Tables 3 and 4. The results are expressed in percent values with respect to the results obtained for Inconel.

Figure 2 shows a specimen at the beginning of the test; after 20 cycless all the materials tested show traces of corrosion on the surface. Figures 3, 4, 5, and 6 show specimens made of AISI 309, AISI 316Ti, AISI 321 and Inconel 625, respectively, after 20 aging cycles.

The results of the tensile characterization (average R_m, R_{p02}, A and Z) as a function of the aging cycles are shown in Figures 7 to 10. The results are presented as normalized values with respect to the results obtained for Inconel at room temperature in the preliminary testing. In Table 4, the maximum percent variations in the values of yield stress

Table 3. Mechanical properties at room temperature.

Material	R_m [MPa]	$R_{p0.2}$ [MPa]	A	Z
AISI 309	0.88	0.86	1.52	2.25
AISI 316Ti	0.80	0.81	1.58	2.20
AISI 321	0.81	0.66	1.75	2.23
Inconel 625	1	1	1	1

Table 4. Mechanical properties at 500ºC.

Material	R_m [MPa]	$R_{p0.2}$ [MPa]	A	Z
AISI 309	0.63	0.46	0.67	0.95
AISI 316Ti	0.69	0.82	0.73	0.93
AISI 321	0.56	0.33	0.61	0.93
Inconel 625	1	1	1	1

Figure 2. Specimen before the ageing test.

Figure 3. Specimen of AISI 309 after 20 ageing cycles.

Figure 4. Specimen of AISI 316Ti after 20 ageing cycles.

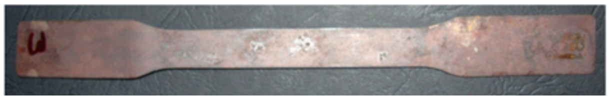

Figure 5. Specimen of AISI 321 after 20 ageing cycles.

Figure 6. Specimen of Inconel 625 after 20 ageing cycles.

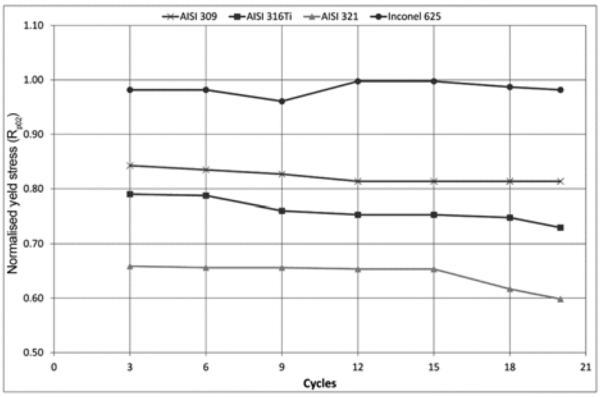

Figure 7. Variation of yield stress R_{p02} during the ageing test.

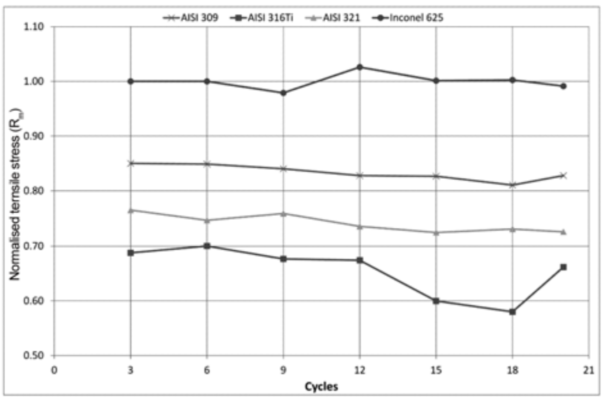

Figure 8. Variation of ultimate tensile stress R_m during the ageing test.

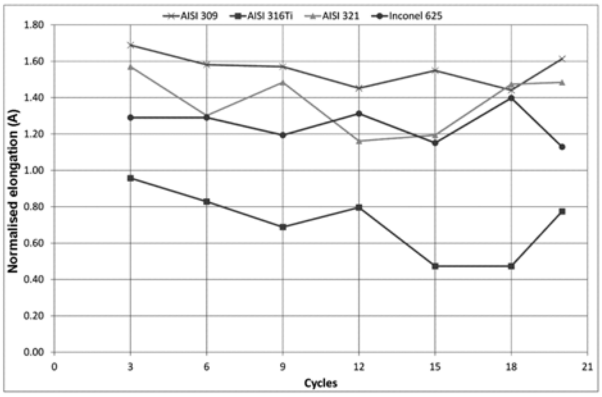

Figure 9. Variation of elongation A during the ageing test.

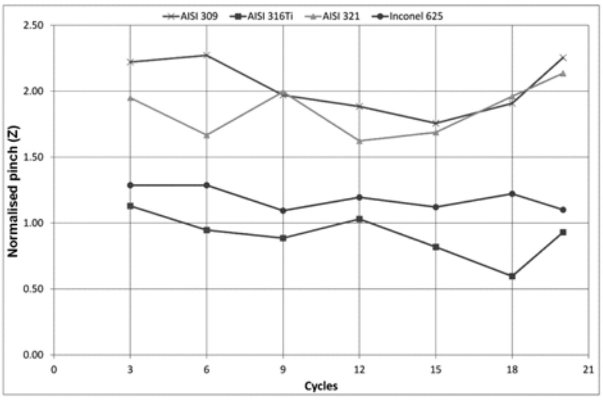

Figure 10. Variation of Z parameter during the ageing test.

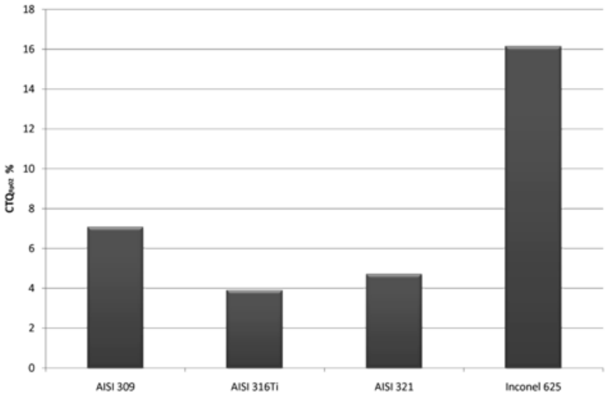

Figure 11. Normalized 0.2% offset yield index $CTQ_{Rp02.}$

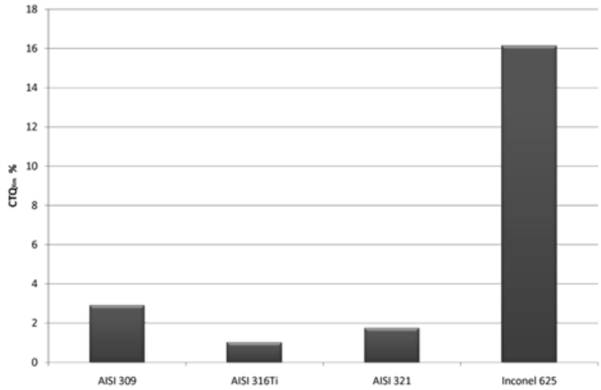

Figure 12. Normalized tensile stress index $CTQ_{Rm.}$

R_{p02}, ultimate tensile stress R_m, elongation A and Z during the test for each material are presented. Again, the results are expressed in percent values with respect to the results obtained for Inconel at room temperature in the preliminary testing.

Table 5. Comparison among the maximum percentage variation of the mechanical properties during the ageing test.

	$\Delta R_{p0.2}$	ΔR_m	Δ(All %)	Δ(Str %)
AISI 309	- 4.9%	- 5.9%	+ 5.33% ; - 2.67%	+ 0.48% ; - 8.86%
AISI 316Ti	- 9.5%	- 27.7%	- 34.33%	- 29.01%
AISI 321	- 9.5%	- 10%	- 14.33%	- 6.49%
Inconel 625	- 3.9%	+ 2.6% ; - 0.9%	+ 12.33%	+ 5.21%

Table 6. Penalty related to corrosion phenomena for each material.

	AISI 309		**AISI 316Ti**		**AISI 321**		**Inconel 625**	
Corrosion	**Danger**	**Entity**	**Danger**	**Entity**	**Danger**	**Entity**	**Danger**	**Entity**
General corrosion	1	0.5	1	0.5	2	2	1	0
Intergranularzcorrosion	1	0.5	1	0.5	2	1	1	0
Pitting corrosion	2	1	1	0.5	3	1.5	1	0
Hot salt corrosion	2	1	1	0.5	2	1	1	0
Penalty	**6**	**3**	**4**	**2**	**9**	**5.5**	**4**	**0**

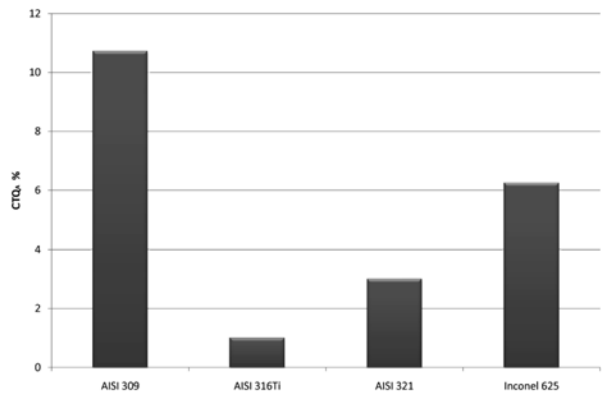

Figure 13. Normalized ultimate elongation index CTQ_A.

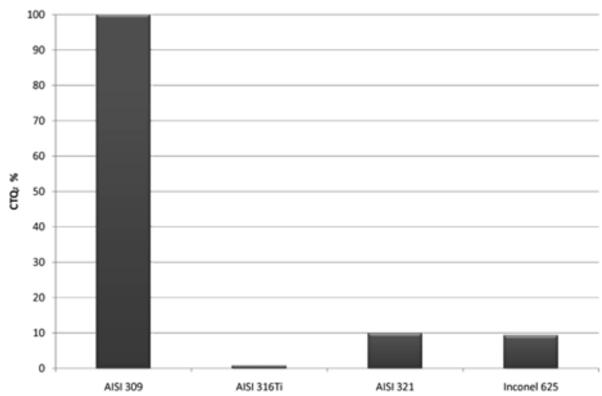

Figure 14. Normalized variation of the specimen Z parameter CTQ_Z.

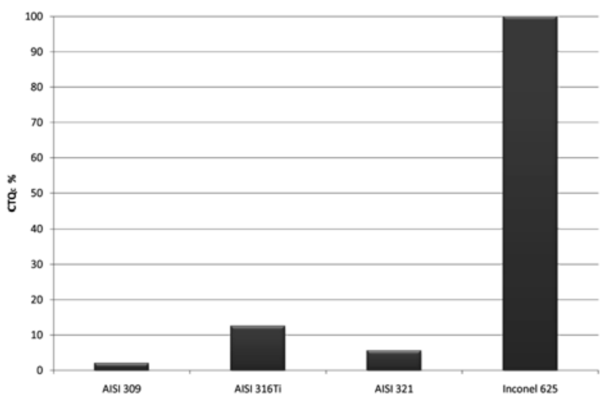

Figure 15. Normalized corrosion index CTQ_C.

Finally, the performance indices have been calculated according to the CTQs approach. Figures 11 to 15 show CTQs for the tensile strength, the 0.2% offset yield, the ultimate elongation, the variation of the specimen width and corrosion, respectively.

Table 6 shows the penalty factors for corrosion for each material according to the parameters shown in Table 2.

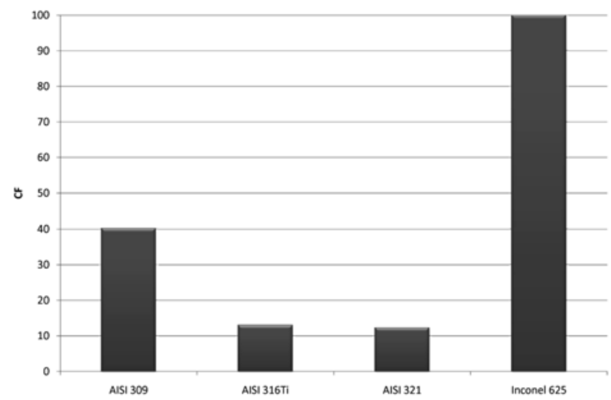

Figure 16. Final ranking of materials.

Figure 16 shows the final ranking of the four materials studied in this work.

4. DISCUSSION

Some observations can be drawn from Figures 7-10 regarding the mechanical behavior of the materials during the aging procedure.

In particular (see Figure 7), the yield stress R_{p02} of both the AISI 309 and AISI 316Ti steels decreases rapidly and steadily as the number of cycles increases. In the case of the AISI 321 steel, the same figure shows a significant decrease in R_{p02} during the final cycles of the aging process (cycles 15 to 21). For the Inconel 625 alloy, the yield stress R_{p02} deceases during the first part of the test, then appears to increase, and finally remains substantially unchanged until the end of the procedure.

Figures 8 shows that the variation in the ultimate tensile stress R_m during the aging test is very minor for the AISI 309 and AISI 321 steels. However, the AISI 316Ti steel shows a decrease in R_m at the end of the aging procedure (cycles 13-18) although the last measured value for R_m appears substantially higher. The Inconel 625 alloy shows an essentially constant ultimate tensile stress value.

As shown in Figure 9, the elongation A shows an irregular behavior for the AISI 309 and AISI 321 steels and for the Inconel 625 alloy. At the end of the test, these three materials have a value of elongation that is not very different from that at the beginning of the test. For the AISI 316Ti steel, the ductility decreases up to 18th cycle, and in the last cycle, the value of elongation increases slightly but remains smaller than that at the beginning of the test.

Figure 10 shows the variation in ductility. Inconel 625 shows a constant value of Z as a function of the number of aging cycles. AISI 321 Ti has a constant behavior until the 12th cycle, and then, the value of Z decreases until the 18th cycle. At the end of the test, this material has a smaller value of Z than that at the beginning of the test. AISI 309 and AISI 321 show irregular behavior, but at the end of the test, AISI 309 has a very similar Z value to that at the beginning of the test, and AISI 321 has a higher Z value than that at the beginning of the test.

Figures 11 to 16 show the comparison of the behaviors of the materials studied in this work, obtained using the CTQs approach. In particular, Figure 11 shows that with regard to the yield stress, Inconel 625 has the highest resistance to the aging test, followed by AISI 309; AISI 321 Ti and AISI 321 have approximately the same scores. With regard to the tensile stress analysis, as reported in Figure 12, Inconel 625 shows better resistance in the aging test, while the other materials present lower CTQs index values. Figure 13 and Figure 14 show the indices for ultimate elongation and the Z parameter, respectively; AISI 309 has the highest scores, followed by Inconel 625, AISI 321 and AISI 321 Ti. The corrosion CTQs index, shown in Figure 15, shows that Inconel 625 is least affected by corrosion phenomena, followed by AISI 309, AISI 321 Ti and AISI 321. The corrosion index has been obtained using the penalties assigned to each corrosion phenomena shown in Table 6. The final ranking of the materials, obtained using the CTQs approach (see Figure 16), shows that Inconel 625 has the highest resistance to the aging procedure, followed by AISI 309. The mechanical properties of AISI 321Ti and AISI 321 are more affected than those of the other materials by the aging test.

The corrosion phenomena that most readily affect the specimen surfaces are pitting corrosion and hot salt corrosion. Figures 17 and 18 show a detailed image (800x magnification) of pitting corrosion and hot salt corrosion, respectively, in an AISI 321 specimen at the end of the test. These phenomena occur as a result of a layer of a mixture of chemicals deposited on the surface during the test. This deposition of chemicals leads to many interstices where breakages occur in the passivation film protecting the surface of the specimen, resulting in corrosion of the metal surface.

The phenomenon of sensitization, which can occur in materials subjected to prolonged conditioning in furnaces,

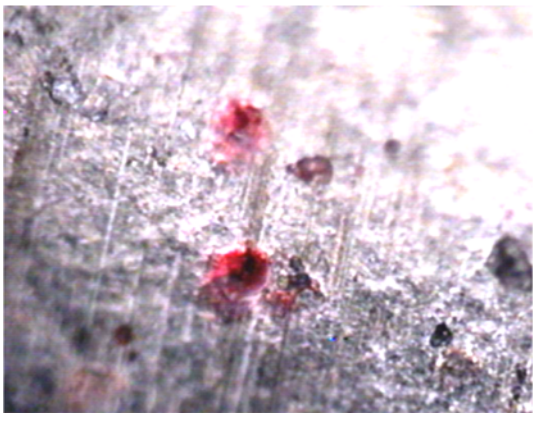

Figure 17. Detail of pitting corrosion on an AISI 321 specimen after 20 ageing cycles (800x magnification).

Figure 18. Hot salt corrosion on an AISI 321 specimen after 20 ageing cycles (800x magnification).

and the consequent risk of intergranular corrosion are avoided in the studied materials because of the presence of high levels of Cr in the AISI 309 steel and stabilizing elements (Ti) in the Inconel 625 alloy and the other austenitic stainless steels.

The results presented in Table 5 show that the austenitic stainless steel AISI 309 and the Inconel 625 alloy are better able to withstand the test conditions; therefore, these two materials are less affected by fatigue and aging phenomena than the other materials.

5. CONCLUSIONS

The experimental procedure developed in this work allows a characterization of four materials that are commonly used in the production of commercial flexible automotive joints.

In particular, the analysis of the variation of all the parameters (ultimate tensile stress R_m, yield stress R_{p02}, elongation A and Z) of the materials during the procedure allows us to draw interesting conclusions about the behavior of all the materials.

It can be observed that the mechanical properties of the Inconel 625 alloy remain almost unchanged, both in terms of strength (tensile stress and yield stress) and in terms of ductility (elongation and ductility), although they have a less regular behavior.

However, the austenitic stainless steel AISI 316Ti is more sensitive to the simulated aging process, showing a significant deterioration in its mechanical properties; in particular, the ultimate tensile stress and the ductility decreased significantly.

In general, it can be concluded that the austenitic stainless steel AISI 309 and the Inconel 625 alloy are less affected than the other two materials by the effects of the aging process during the testing.

The analysis of the specimen surfaces, after several cycles of aging, shows corrosive phenomena such as pitting corrosion and hot salt corrosion, which occur as result of the breakage of the passivation film.

The Inconel 625 alloy shows a significantly higher resistance to corrosion compared with the stainless steels. However, the cost of the Inconel 625 is significantly higher than that of austenitic steels; the cost of the Inconel 625 is a significant obstacle to its application in commercial automotive components.

The phenomenon of sensitization (precipitation of carbides on grain), which can occur as a result of prolonged conditioning at high temperature, and the consequent risk of intergranular corrosion are avoided in the studied materials because of the presence of certain substances, such as a high levels of Cr in AISI 309 and stabilizing elements (Ti) in the Inconel 625 alloy and the other austenitic stainless steels.

The materials' characteristics have been also compared using the CTQs approach. CTQs results confirm that Inconel 625 is the material that best resists the conditions simulated by the aging procedure developed in this work.

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