# EFFECT OF AGING ON FRACTURE TOUGHNESS PARAMETERS AND TENSILE PROPERTIES OF 17-4 PH STAINLESS STEEL.

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17-4 PH stainless steel in the solution-treated condition has been aged for 1 hour in the range of temperatures between 480 °C and 600 °C, with different cooling rates. The thermal aging treatment promotes the precipitation hardening of the alloy that strongly influences the microstructure and, as a consequence, the fracture toughness and the tensile properties.  $K_{IC}, J_{IC}$  and Charpy V-notch impact tests have been performed on the aged specimens. An increase of the aging temperature results in an increase in toughness values.

## INTRODUCTION

17-4 PH stainless steel (AISI 630) is widely used as a structural material in a variety of applications in aircraft, chemical and nuclear industries, for the excellent mechanical properties combined with good corrosion resistance. In order to achieve the optimum combination of tensile properties, toughness and impact strength the alloy is subjected to the heat treatment consisting of homogenisation at about 1040 °C, air cooling to room temperature and aging between 480 °C and 600 °C (1, 2). The aim of this work is to study the influence of aging treatments on the microstructure and on the strength and fracture toughness properties of 17-4 PH stainless steel.

## EXPERIMENTAL PROCEDURES

The chemical composition of the alloy is given in Table 1.

TABLE 1 - Chemical composition of the investigated 17-4 PH (wt. %)

C	S	P	Si	Mn	Cr	Ni	Mo	Cu	Sn	Nb
0.049	0.005	0.021	0.26	0.62	15.73	4.40	0.23	3.36	0.006	0.28

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After the solution treatment at 1040 °C for 1 h, followed by air cooling, the samples were aged at 480-510-540-600 °C for 1 h and air cooled. In order to evaluate the influence of the cooling rate on the microstructural and mechanical properties, the Charpy specimens were also subjected to both air cooling and water-quenching after the aging treatment.

The corresponding tensile and fracture toughness parameters were established using:

a) tensile test specimens (according to ASTM E 8M)

b) Charpy V-notch (CVN) specimens (according to ASTM E 23)

c) compact tension specimens (according to ASTM E 399)

#### RESULTS

#### Microstructure

The microstructure of the unaged material consists of martensite (Fig.1a) with occasional presence of  $\delta$ -ferrite phase, elongated in the prior working direction. In the heat-treated condition, the alloy also contains some retained austenite.  $\delta$ -ferrite phase fraction varied from 5 to 10% and austenite grain size between 25 to 35  $\mu$ m.

Figure 1 shows the microstructure of the alloy aged for 1 h at different temperatures. Thermal aging between 480 °C and 510 °C probably produces a precipitation of  $M_{23}C_6$ -type particles (3) at the lath boundaries of martensite. The formation of a f.c.c. copper-rich phase is also reported for 17-4 PH steel at these aging temperatures (4,5,6). On the specimens aged for 1 h between 540 °C and 600°C the transformation of martensite is almost completed. The alloy aged at 600 °C for 1 h shows also a certain amount of austenite predominantly along the lath boundaries.

#### Mechanical Properties

The mechanical properties of this alloy are very sensitive to the aging temperature, because of the direct relationship with the precipitation processes. The investigations on the precipitation sequence in the 17-4 PH stainless steel are based on the assumption that the process of precipitation is the same as that observed in Fe-Cu and Fe-Cu-X alloys (4,5). Age hardening involves initial formation of coherent copper-rich clusters which transform to incoherent f.c.c. ε-Cu precipitates on prolonged aging. In this condition the alloy shows a sharp increase in the hardness values with a maximum between 480 °C and 510 °C (Fig.2a).

Impact Tests. Impact energies, measured with an instrumented pendulum, show a minimum value after aging at 480 °C for 1 h (Fig.2b) which is primarily due to a minimum in the crack propagation energy; the crack nucleation energy is less sensitive to

aging (Fig.3a). On increasing the aging temperature there is a substantial improvement in the fracture toughness.

This behaviour has been explained by a difference on the grain boundary concentration of phosphorous (7,8,9). At lower aging temperatures the phosphorous segregation causes temper embrittlement of the alloy. The phosphorous segregation is also responsible for the appreciable difference in hardness values and impact toughness observed in specimens subjected to different cooling practices after aging, for all the aging temperatures. The water-quenched specimens, with minor phosphorous concentration at grain boundary, exhibit hardness values less than air-cooled ones (Fig.2a) and a substantial improvement in impact toughness (Fig.2b).

<u>Tensile Tests.</u> The increase of the aging temperature, from  $480\,^{\circ}\mathrm{C}$  to  $600\,^{\circ}\mathrm{C}$ , results in a continuous reduction of the tensile properties of the 17-4 PH steel (Fig.4a). On the contrary the growth of the aging temperature improves the ductility of the steel in terms of reduction of area and percentage elongation (Fig.4b).

Toughness Tests. The trend evidenced by tensile tests has been confirmed by K<sub>IC</sub> tests performed on CT-1 samples; toughnesses are relatively low at the aging temperature of 480 °C and seem to reach maximum values in the range of temperatures between 540 °C and 600 °C (open square symbols in Fig.3b). All the specimens broke suddenly just after the first straight linear portion of the load-load point displacement plot. Valid K<sub>IC</sub> values have been obtained for all the aging temperatures. Modified CT specimens have been utilized to measure displacement along the loading line; tests have been controlled by a computer HP 300 series 9000; the area under the loaddisplacement curve up to the point utilized to determine K<sub>IC</sub> (generally corresponding to about 2% crack advance) has been measured. From these area measurements approximate values of  $\ensuremath{\mathrm{J}}_{\ensuremath{\mathrm{C}}}$ can be obtained by the usual ASTM E 813-81 formula by utilizing the original crack lengths (ao) of the specimens. KC data derived by JC values under plane strain conditions (cross symbols in Fig.3b) substantially confirmed K<sub>IC</sub> results.

Fractography

The fracture appearance is strictly related to heat treatment and stress state. SEM analyses of the fracture surfaces of the Charpy specimens has revealed a good correlation between the impact values and the relative contributions of ductile and brittle failure modes. The solution-treated specimens show relatively uniform dimples, the larger being related to the presence of Nb and Cr carbides (Fig.5a). The specimens aged between 480 °C and 510 °C show large amounts of quasi-cleavage failure (Fig.5b). Increasing the aging temperature,

resulted in specimens failing predominantly by a process of void coalescence (ductile fracture). In this condition the fracture surface exhibits smaller and highly nonuniform dimples (Fig.5c). In all cases fracture next to the notch starts immediately in a ductile manner.

## CONCLUSIONS

The microstructure and mechanical properties of the 17-4 PH steel are very sensitive to the influence of the aging parameters (temperature and cooling rate). The range between 480 °C and 600 °C has been considered.

- 1. Impact energies show a minimum for the steel aged at 480 °C for 1 h. The influence of the cooling rate on CVN energy values has been investigated: the water-quenched specimens exhibit an improvement on toughness compared with the air-cooled ones due to the different phosphorous segregation at grain boundaries.
- 2. Higher tensile properties result at the lower aging temperatures. 3. Standard toughness tests show the same behaviour of the impact tests. This trend is in agreement with the precipitation sequence induced by the aging treatment.

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#### REFERENCES

- Ravez, R., Trait. Therm., Vol. 134, 1979, pp. 55-59. (1)
- Woulds, M., Lloyd, R., Precis. Met., Vol. 40, 1982, pp. 62-65. Antony, K.C., J.Met., Vol.15, 1963, pp.922-927. (2)
- (3)
- Youle, A., Ralph, B., Met. Sci. J., Vol.6, 1972, pp. 149. (4)
- Hornbogen, E., Acta Met., Vol.10, 1962, pp.525. (5)
- Rack, H.J., Kalish, D., Met. Trans., Vol.5, 1974, pp.1595-1605. (6)
- Misra, R.D.K., Prasad, C.Y., Balasubramanian, T.V., Rama Rao, P., Scripta Met., Vol.20, 1986, pp. 713-716.
- Misra, R.D.K., Prasad, C.Y., Balasubramanian, T.V., Rama Rao, P., Scripta Met., Vol.21, 1987, pp. 1067-1070.
- Misra, R.D.K., Prasad, C.Y., Balasubramanian, T.V., Rama Rao, P., "ICF 7. Advances in Fracture Research", Vol.4, Houston, Texas, Edited by Pergamon Press, Headington, Hill Hall, Oxford, UK, 1989.
- Wagner, D., Chavaillard, J.-P., Meyzaud, Y., Bonningue, M., Cozar, R., Baudry, G., J. Mat. Energy Systems, ASM, Vol. 6, 1985, pp. 263-272.

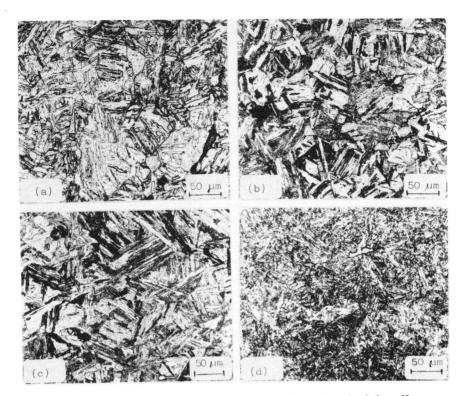


Figure 1. Microstructure of the unaged alloy (a) and of the alloy aged for 1 h at  $\,480~^{\circ}C$  (b),  $\,540~^{\circ}C$  (c),  $600~^{\circ}C$  (d).

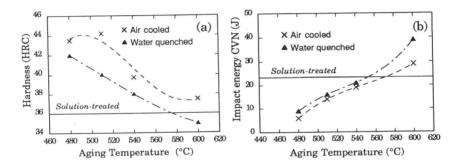


Figure 2. Variation of the hardness values (a) and of the impact toughness (b) with aging temperatures.

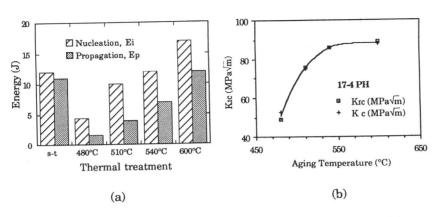


Figure 3. (a) Nucleation and propagation energies obtained by Charpy tests. (b) Measured  $K_{IC}$  values (\*);  $K_C$  data derived trough  $J_C$  measurements (+)

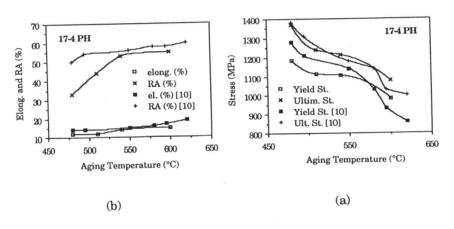


Figure 4. Comparison between (a) tensile properties, (b) % elongation and reduction of area of the 17-4 PH steel considered (x, x) and data from bibliography (x, +)

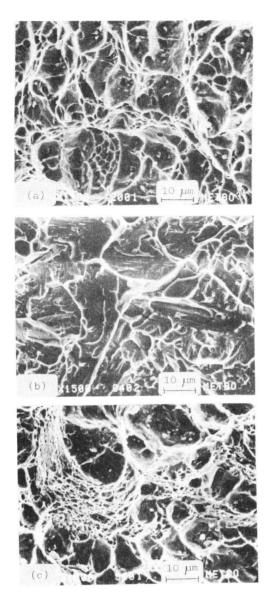


Figure 5. SEM micrographs of the fracture surfaces of Charpy specimens: (a) solution-treated alloy, (b) samples aged at 480 °C, (c) samples aged at 600 °C.