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EFFECT OF AGEING A TWO-PHASE Fe-NiCrMo ALLOY ON THE STRAIN HARDENING AT ROOM TEMPERATURE AND AT 290 °C

VPLIV STARANJA DVOFAZNE ZLITINE Fe-NiCrMo NA DEFORMACIJSKO UTRJEVANJE PRI SOBNI TEMPERATURI IN PRI 290 °C

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Fe-NiCrMo alloys were aged in the temperature range 290 °C to 350 °C for the spinodal decomposition of the ferrite solid solution. The tensile properties and the Charpy notch toughness were determined at room temperature and at 290 °C. The stress-strain curves were examined and the strain-hardening exponent was determined. This exponent changed little during the increase of the ferrite hardness and it is greater at 290 °C than at room temperature. This confirms the explanation proposed for the different effect of the test temperature on the notch toughness and the tensile properties.

Key words: austenite-ferrite microstructure, mechanical properties, ferrite hardness increase, strain hardening

Zlitina Fe-NiCrMo je bila starane v razponu temperature od 290 °C do 350 °C za spinodalno razgradnjo trdne raztopine v feritu. Določene so bile raztržne lastnosti in zarezna žilavost pri sobni temperaturi in pri 290 °C. Analizirane so bile krivulje napetost – deformacija in določen eksponent deformacijske utrditve avstenita. Ta eksponent se malo spremeni zaradi povečanja trdote ferita in je večji pri 290 °C kot pri sobni temperaturi. To potrjuje razlago, zakaj so pri višji temperaturi raztržne lastnosti nižje, zarezna žilavost pa večja kot pri sobni temperaturi.

Ključne besede: mikrostruktura ferita in avstenita, mehanske lastnosti, povečanje trdote ferita, deformacijska utrditev

1 AIM OF THE INVESTIGATION

During the ageing of Fe-NiCrMo alloys with a microstructure of austenite and ferrite the hardness of the ferrite is increased because of the spinodal decomposition of the solid solution in ferrite into two constituents: one enriched in chromium and the second enriched in nickel. In both phases the initial α-iron lattice is preserved, while the lattice parameters are modified. Both constituents accommodate with elastic internal stresses that increase the hardness and brittleness and, after magnetisation, result in alloys with a suitable chemical composition and hard magnetic properties[1,2]. The kinetics of the decomposition depends on the diffusional transport of atoms in the substitutional solid solution in the ferrite. The rate of diffusion depends strongly on the temperature, and for this reason, the rate of spinodal decomposition-ageing is very slow in the temperature range in which alloys of this type are operating in power plants. In the ageing process of ferrite, the solid solution in austenite is stable; it may only change with the precipitation of carbides if the content of carbon is above the solubility limit. At higher temperature, the spinodal decomposition is replaced by the formation of σ-phases, which greatly diminishes the ductility of the alloys[3]. With ageing, the properties of the alloys with a two-phase microstructure of austenite and ferrite are changed, depending on the volume share of ferrite and the extent of its decomposition.

In investigations of this type of alloys[4] it was established that the change of properties was greater for an alloy with 14.5 % of δ-ferrite than for an alloy with 8.5% of δ-ferrite when ageing in the temperature range 300 – 400 °C, while at higher temperatures the ageing effect was smaller. These findings were confirmed[5], where it was found, also, that the content of δ-ferrite determined from the Schaeffler diagram was unreliable and that the distribution of ferrite in as-cast alloys was inhomogeneous and could vary between 1.5 and 22.5 for the same cast piece. The ageing effect on the Charpy toughness was very strong in the temperature range 303 °C to 325 °C and the initial toughness was achieved again after annealing at 550 °C. Of the several processes that could affect the alloy's properties, the main embrittlement process is that of spinodal decomposition[6]. A correlation was developed[7] for the assessment of the embrittlement and the prediction of changes in the fracture and the Charpy and tensile properties of the as-cast two-phase alloys. The use of small specimens for the investigation of in-service-aged elbows gives reliable values for the J-Δa results on the condition that strongly deviating specimens are rejected[8]. The low-cycle fatigue increases rapidly with the increase of the ageing time[9]. In[10,11] these findings were confirmed with the investigations of alloys with a different volume share of ferrite.

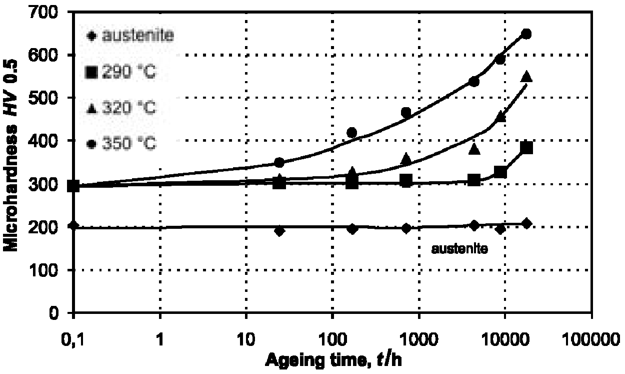

Figure 1: Effect of ageing time at 290 °C, 320 °C and 350 °C on the micro-hardness of the ferrite and austenite

Slika 1: Vpliv časa staranja pri 290 °C, 320 °C in 350 °C na mikrotrdoto ferita in avstenita

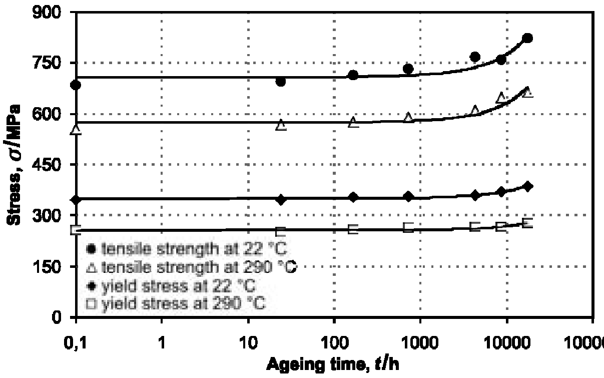

Figure 3: Effect of ageing time on the yield stress and the tensile strength at room temperature and at 290 °C

Slika 3: Vpliv trajanja staranja na mejo plastičnosti in raztržno trdnost pri sobni temperaturi in pri 290 °C

It was found, also, that the tensile properties of the unaged and aged alloys were lower and that the notch toughness was higher when testing at 290 °C. The aim of this work was to establish whether the difference was related to the different strain-hardening behaviour of the alloys at room temperature and at 290 °C.

In **Figure 1** the effect of the ageing time and temperature on the ferrite and austenite microhardness are shown[10,11]. The austenite micro-hardness is not affected by the ageing, while the micro-hardness of the ferrite starts to increase after an ageing time that is shorter with higher temperature when the increase of the hardness is faster and stronger.

In **Figures 2 and 3** the tensile properties and the notch toughness are given as a selection of data in[10,11]. After ageing and a hardness increase of the ferrite from HV 295 to about HV 650 units (an increase of the hardness by 2.15 times), the elongation and the reduction of area are decreased at both testing temperatures by about 30 %. For the average of the specimens aged for different times, the elongation and the reduction of area are lower by about 25 % at 290 °C than at room temperature.

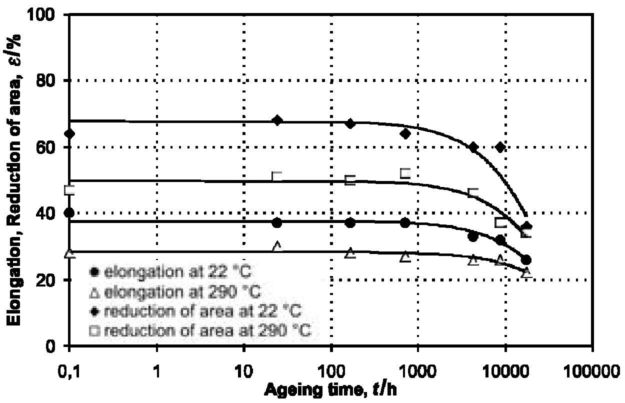

Figure 2: Effect of ageing time on the elongation and the reduction of area at room temperature and at 290 °C

Slika 2: Vpliv trajanja staranja na razteznost in kontrakcijo pri sobni temperaturi in pri 290 °C

The yield stress is increased very slightly with the increase of the ferrite hardness at both testing temperatures, and it is only about 11 % greater for both testing temperatures after the hardness of the ferrite was increased to HV 650 units. For the tensile strength, the effect of ageing time is greater, as at both testing temperatures it is increased by about 18 %. The effect of the testing temperature is greater for the yield stress, as it is decreased on average by 27 % at 290 °C, while the tensile strength is decreased by about 19 %. The changes are, in absolute values, larger with a large initial level and are of 99 MPa for the yield stress and 140 MPa for the tensile strength.

In **Figure 4** the effect of ageing time at three temperatures on the notch toughness is shown for testing at room temperature and at 290 °C. When testing at room temperature, the notch toughness starts to decrease faster after ageing at 350 °C and 320 °C after the ferrite hardness has increased to about 350 units. This shows that the notch toughness is more sensitive to the effect of the ferrite's hardness increase. In[11] an explanation was proposed; that in the process of Charpy plastic deformation and fracturing during a rapid toughness decrease, the ferrite starts to fracture with a cleavage and that the accelerated decrease of the notch toughness is related to the cleavage of the ferrite inserts ahead of the tip of the propagation fracture. By ageing at 350 °C at a ferrite hardness of about 540 units a level of notch toughness is achieved that is not changed during any further increase of the hardness to 650 units. With a lower ageing temperature the final notch toughness level is achieved after the longest ageing time. The explanation could be either a difference in the extent of the spinodal decomposition due to the difference in the ageing temperature or that due to a level of hardness of about 450 units. It is mostly those ferrite inserts fracture with cleavage that have a favourable space orientation with regards to the plane of the propagating crack.

For the non-aged alloy the notch toughness is higher at 290 °C than at room temperature by about 20 J or 18

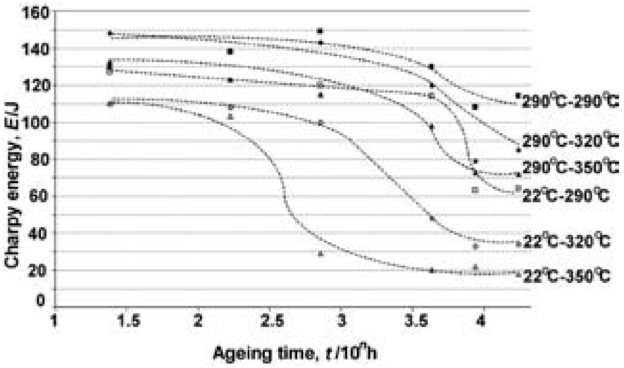

Figure 4: Effect of ageing time at three temperatures on the notch toughness at room temperature and at 290 °C
Slika 4: Vpliv časa staranja pri treh temperaturah na zarezno žilavost pri sobni temperaturi in pri 290 °C

%. After the longest ageing time the notch toughness at room temperature is decreased by about 82 % and much less, only by about 36 %, at a temperature of 290 °C. Accordingly, at this stage of the decomposition of the solid solution in ferrite, the difference in the notch toughness at 290 °C and room temperature is increased by about 3.5 times.

The tensile properties are lower and the notch toughness is higher when testing at 290 °C, than at room temperature. In[11] it is assumed that the differences between the properties at room temperature and 290 °C are probably the difference in the deformation and the fracturing behaviour of both kinds of tests due to the presence of the notch on the toughness specimens and the difference in test time, which cause a high local heating due to the very fast deformation and fracturing during the testing of the notched specimens[12]. A possible effect of the different strain hardening at both testing temperatures is surmised and this investigation is aimed at its experimental verification. The ageing temperature of 350 °C was selected because it results in the largest and fastest changes of ferrite hardness and notch toughness.

2 EXPERIMENTAL WORK

For this investigation the alloy and specimens in[10,11] were used, although the details on the preparation, ageing and testing of the alloys will also be summarised here. The composition of the investigated alloy was: 0.06 C, 1.68 Si, 0.67 Mn, 0.03 P, 0.01S, 9.0 Ni, 20.8 Cr and 2.46 Mo. The microstructure of the alloy is shown in **Figure 5.** The ageing temperatures were 290 °C, 320 °C and 350 °C and the ageing time was up to 17.520 h. Only specimens aged at 350 °C were used in this investigation because of the highest increase of the ferrite hardness. All the mechanical tests were performed at room temperature (22 °C) and at 290 °C. The micro-hardness was determined only at room temperature. Additional measurements of the ferrite micro-hardness were performed to determine more accurately the hardness increase during the ageing time of the start of the rapid decrease of the notch toughness and the tensile tests were performed for specimens aged for up to the longest ageing time. Austenite does not fracture with a cleavage, and so for this reason, and according to[8], the exceedingly deviating Charpy results were rejected as being due to the local, greatly increased content of ferrite in the specimen with a small section and the as-solidified microstructure.

The uniform plastic deformation was determined by measuring the lengthening of the strain on the stress-strain curves from the end of the elastic deformation to the maximum stress of the strain curves. This part of the true-stress, true-strain curve was digitalised, a linear dependence was obtained in the log-log coordinates and the approximate strain coefficient was deduced from its slope for specimens aged for different times at 350 °C.

The shape of the recorded stress-strain curves in **Figures 6** and **7** shows that the tensile behaviour of the alloy depends on the extent of the age hardening of the ferrite and of the testing temperature. In **Figures 8** and **9** the strain hardening part of the stress-strain curves between the start of the plastic deformation and the maximum tensile force are shown. The linear shape confirms that the slope of the dependence can be used as a reliable representation of the strain-hardening exponent.

Theoretically, the effect of increasing the strain on the deformation resistance is due to the increase in the number of dislocations, and it is deduced as[13]

$$\sigma_\varepsilon - \sigma_0 = \alpha \text{ G b } \rho^{1/2} \qquad (1)$$

where σ is the deformation resistance at a deformation of ε, σ_0 is the deformation resistance at the start of the plastic strain, ρ is the density of dislocations, G is the shear modulus and b is the Burgers vector.

The equation is not used because of the experimental complexity of determining the density of the dislocations. The strain-hardening exponent is used instead of

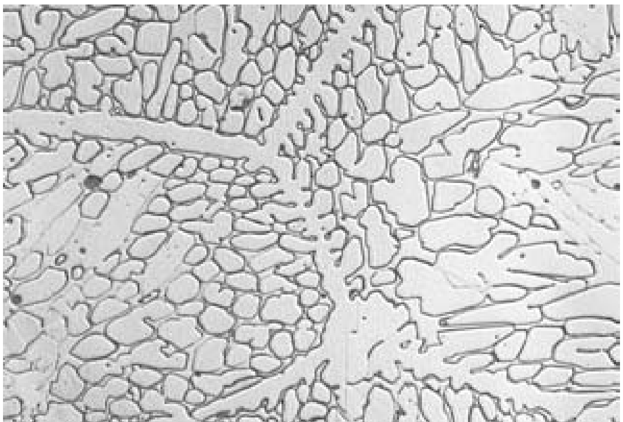

Figure 5: Microstructure of the investigated alloy with 27 % of ferrite
Slika 5: Mikrostruktura raziskovane zlitine s 27 % ferita

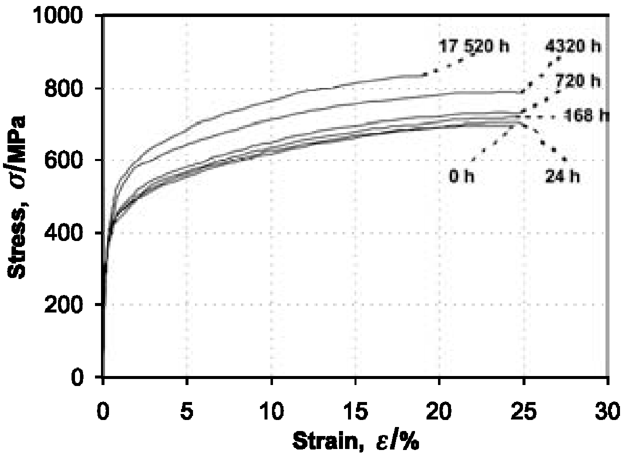

Figure 6: Strain-stress curves at room temperature for specimens aged for different times at 350 °C

Slika 6: Odvisnost napetost – deformacija pri sobni temperaturi za preizkušance, ki so bili starani različno dolgo pri 350 °C

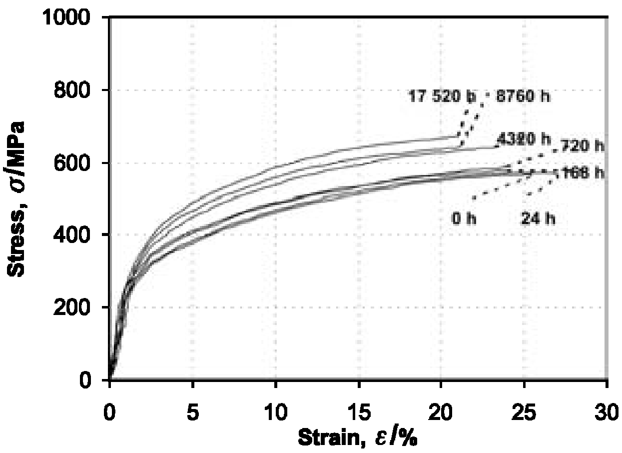

Figure 7: Strain-stress curves at 290 °C for specimens aged for different times at 350 °C

Slika 7: Odvisnost napetost – deformacija pri 290 °C za preizkušance, ki so bili starani različno dolgo pri 350 °C

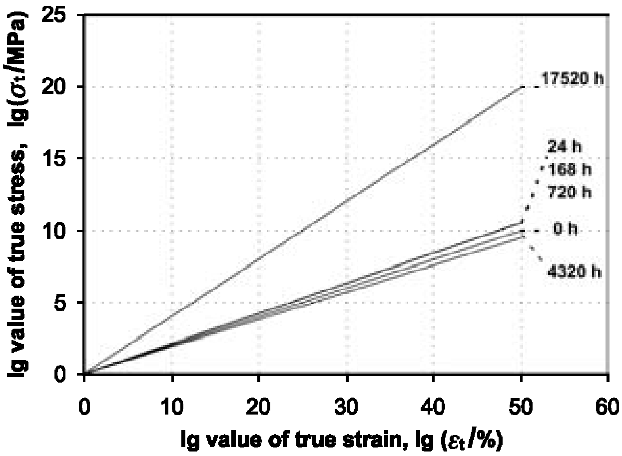

Figure 8: True-strain, true-stress dependence for the strain-hardening part of the curves in **Figure 6**

Slika 8: Odvisnost prave napetosti od prave deformacije za del krivulj na **sliki 6** z deformacijsko utrditvijo v logaritemskih koordinatah

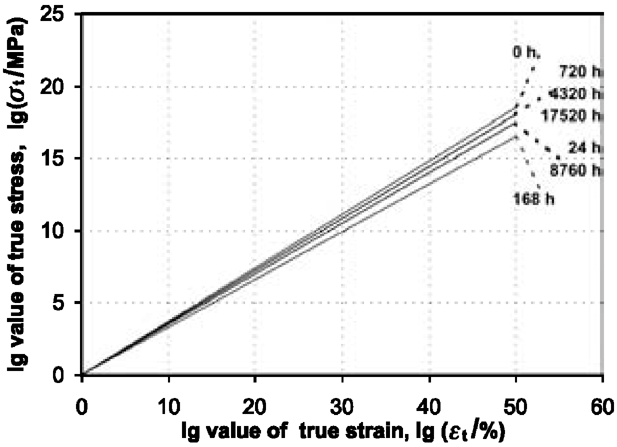

Figure 9: True-strain, true-stress dependence for the strain-hardening part of the curves in **Figure 7**

a measure of the effect of the plastic strain on the deformation resistance. It is deduced from equation[14]:

$$\sigma_\varepsilon = k(\varepsilon_0 + \varepsilon)^n \qquad (2)$$

where k is a constant, ε_0 is the initial strain, ε is the strain increase, and n is the strain-hardening exponent.

For tensile tests k is a constant, and $\varepsilon_0 = 0$ if the strain hardening is started at the end of elastic deformation. In our case equation (2) can be written in the form $\sigma = k\,\varepsilon^n$, which also describes the dependences in **Figures 8** and **9**. Since the value of the strain-hardening exponent was established from tests in equivalent conditions it can be used as a comparative value.

3 DISCUSSION

In **Tables 1** and **2** the numerical data established from the stress-strain curves and the ferrite hardness are given. The ratio of the proportion to the maximum stress is, at room temperature, independent on the increase of ferrite hardness, while it increases slowly with this hardness at 290 °C. The plastic extension, a measure of the plastic strain before the start of the reduction of area, decreases very slowly with the increasing hardness of ferrite at both testing temperatures. In contrast, the approximate exponent of the deformation hardening is, for the ferrite hardness up to approximately HV 537 units, significantly lower at room temperature than at 290 °C. Above this hardness level it increases up to a value similar to that for testing at 290 °C. No similarity was found between the effect of the increase of the ferrite hardness on the coefficient of strain hardening and the notch toughness, as the toughness is greatly diminished by an unchanged strain-hardening coefficient. On the other hand, the coefficient of strain hardening is higher with a higher notch toughness at 290 °C than at room temperature. The notch toughness for ductile fracturing also depends strongly on the extent of the deformation before the crack is started at the notch tip[15]. All the tensile properties are higher at room temperature

Table 1: Characteristic parameters of the stress-strain curves in **Figures 6** and **8**. Testing at room temperature, ageing temperature 350 °C
Tabela 1: Karakteristični parametri iz odvisnosti napetost deformacija na **slikah 6** in **8**. Preizkus pri sobni temperaturi, temperatura staranja 350 °C

Ageing *t*/h	E_{pr}/MPa	E_m/MPa	E_{pr}/E_m	Plastic extension	*n*	Ferrite hardness *HV*
0	341	708	0.48	0.27	0.20	295
24	362	703	0.51	0.25	0.21	350
168	356	720	0.49	0.24	0.21	419
720	352	729	0.49	0.25	0.21	465
4320	371	790	0.47	0.24	0.19	537
17520	376	832	0.47	0.19	0.40	648

Table 2: Characteristic parameters of the stress-strain curves in **Figures 7** and **9**. Testing at room temperature, ageing temperature 350 °C
Tabela 2: Karakteristični parametri iz odvisnosti napetost deformacija na **slikah 6** in **7**. Preizkus pri sobni temperaturi, temperatuira staranja 350 °C

Ageing *t*/h	E_{pr}/MPa	E_m/MPa	E_{pr}/E_m	Plastic extension	*n*	Ferrite hardness *HV*
0	268	567	0.47	0.24	0.37	295
24	268	572	0.47	0.24	0.35	350
168	278	583	0.48	0.23	0.33	419
720	299	573	0.52	0.22	0.36	465
4320	317	646	0.49	0.22	0.36	537
8760	327	656	0.50	0.23	0.35	589
17520	350	675	0.52	0.19	0.36	648

E_{pr} is the stress at the end of proportionality $\varepsilon - E$
E_m is the maximum stress before the start of the reduction of area
Plastic extension is the extension between the points E_{pr} and E_m
n is the approximate exponent of strain hardening

than at 290 °C and, as only the exponent of strain hardening is higher at 290 °C, the assumption is confirmed that the higher notch toughness is related to the share of energy consumed before the crack initiation at the notch tip,[11] which is greater at 290 °C than at room temperature. The difference reflects the effect the difference in the shape of the notch test specimen and the loading geometry, the asymmetrical flexion for the notch toughness test and the axial-symmetry for the tensile test.

4 CONCLUSIONS

With the analysis of the stress-strain curves for specimens with a duplex austenite-ferrite microstructure and different ferrite hardnesses obtained with tests at room temperature and 290 °C it was established that with a higher temperature the strain hardening of the austenite matrix is greater than at room temperature. This finding is in agreement with the explanation that the greater share of energy consumed before the crack is opened at the notch tip is the cause of the higher notch toughness at 290 °C, although the tensile properties are higher at room temperature.

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