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EFFECT OF TEMPERING ON THE ROOM-TEMPERATURE MECHANICAL PROPERTIES OF X20CrMoV121 AND P91 STEELS

VPLIV POPUŠČANJA NA MEHANSKE LASTNOSTI JEKEL X20CrMoV121 IN P91 PRI SOBNI TEMPERATURI

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The effect of tempering time and temperature on the room-temperature tensile properties and hardness of two martensitic creep-resistant steels, X20CrMoV121 and P91, was investigated. Samples cut from industrial tubes were tempered for 17520 h at 650 °C and for 8760 h at 750 °C. On the tempered samples the yield stress, tensile strength, and hardness at room temperature were determined and an SEM examination was carried out.

It was found that the effect of tempering at 750 °C on the microstructural changes, room-temperature tensile properties and hardness was greater for both steels than the effect of tempering at 650 °C. The changes in the yield stress, tensile strength and hardness of both steels at a given tempering temperature were found to be very similar. Therefore, a general mathematical expression with specific coefficients for each property was deduced. These results are part of a larger investigation aimed at establishing a correlation between the particle spacing, yield stress, creep rate and hardness, which could be useful in an evaluation of the lifetime issues relating to the thermal-power-plant components.

Keywords: tempering, microstructure, mechanical properties, X20CrMoV121 and P91 steels

Vpliv časa in temperature popuščanja na raztržne lastnosti in trdoto pri sobni temperaturi je bil raziskan pri martenzitnih jeklih X20CrMoV121 in P91, ki sta odporni proti lezenju. Preizkušanci so bili izrezani iz industrijskih cevi in popuščeni do 17520 h pri 650 °C in 8760 h pri 750 °C. Na popuščenih vzorcih so bile določene meja plastičnosti, raztržna trdnost in trdota pri sobni temperaturi, mikrostruktura pa preiskana v SEM.

Ugotovljeno je bilo, da je vpliv popuščanja pri 750 °C na spremembo mikrostrukture, raztržne lastnosti pri sobni temperaturi in trdoto večji pri obeh jeklih, kot vpliv popuščanja pri 650 °C. Spremembe meje plastičnosti, trdnosti in trdote so bile podobne pri obeh jeklih pri dani temperaturi popuščanja. Razvita je bila zato matematična odvisnost s specifičnimi koeficienti za vsako lastnost. Rezultati so del širše raziskave, katere cilj je opredeliti korelacije med razdaljo med izločki, mejo plastičnosti, hitrostjo lezenja in trdoto, ki bi bile koristne pri oceni preostale trajnostne dobe komponent termoelektrarn.

Ključne besede: popuščanje, mikrostruktura, mehanske lastnosti, jekli X20CrMoV121 in P91

1 INTRODUCTION

In recent years there has been an increased demand to improve the efficiency of steam power plants for economical and environmental reasons.[1–4] A straightforward way to achieve this is to raise the inlet temperature and pressure of the steam that passes through the turbines. This directly saves the fuel and reduces the CO_2 emissions.[5]

The problems with higher steam temperature and pressure are largely material related. The microstructures of the materials operating under such conditions change with time and, consequently, several degradation mechanisms such as creep, fatigue, thermal fatigue, creep-fatigue, progressive embrittlement, corrosion/oxidation, etc., are accelerated. Among these damage mechanisms, the most important are the damages caused by an increase in the creep deformation. The main candidate materials for building the plants with more advanced steam parameters are 9–12 % chromium steels.[6, 7]

The risk of a failure due to creep deformation and other damage mechanisms is always present. Therefore, periodical checking of their properties and residual lifetime after a determined period of operation of the power plants is always necessary. The checking of the creep rate and the creep strength is expensive and time consuming. For this reason, simpler methods using faster and less expensive tests that make it possible to identify the changes in the properties of the steels already employed in the vital parts of a power plant, have been developed. One of these methods is checking the room-temperature mechanical properties and the microstructure after a certain tempering time, simulating the changes in the microstructure and the properties that occur after longer operation periods (in real conditions). It has been shown recently that the time when the creep failure occurs is related to the yield stress and the tensile strength at creep temperature[8,9] and that hardness is related to creep life.[10] It was also shown[11] that within a certain range of the room-temperature yield stress (350 MPa to 650 MPa) the accelerated creep rate at 580 °C decreases continuously from $8 \cdot 10^{-7}$ s^{-1} to $5 \cdot 10^{-9}$ s^{-1}.

2 EXPERIMENTAL WORK

In the present work, the X20CrMoV121 and P91 steels were chosen for an investigation. The samples were cut from the pipelines with ϕ = 38 mm × 8 mm and ϕ = 82 mm × 14.5 mm. The quantometer chemical compositions for both steels are given in **Table 1**.

Before extracting the specimens for the room-temperature tests and examinations, the samples of both steels were tempered for discrete times up to 17520 h at 650 °C and for a shorter time up to 8760 h at 750 °C to simulate the changes in the microstructure that take place under real operating conditions in the power plants, and their effect on the room-temperature tensile properties and hardness.

Static-tensile tests at room (ambient) temperature were performed on the specimens extracted from the previously tempered samples. The tests were initially performed on the specimens prepared from the as-delivered tubes and then on the specimens tempered for 2 h, 4320 h and 8760 h at 650 °C and 750 °C, and up to 17520 h at 650 °C. All the tensile tests were performed on a 500-kN static-dynamic testing machine in the Laboratory for Mechanical Testing at the Institute of Metals and Technology. A part of these results was published in the proceedings of IPSSC.[12]

With the aim to assess the changes in the microstructure as a function of tempering time and temperature, the SEM specimens were prepared with the standard metallographic techniques.

A Jeol – JSM6500F Field Emission Scanning Electron Microscope (FE-SEM) was used to acquire images at three different magnifications, namely, 2000-, 5000- and 10000-times, with the working parameters of the 15-kV acceleration voltage, 7-nA probe current and 10-mm working distance. Images were acquired from the specimens in the initial (as-delivered) state and from those tempered for 2 h, 4320 h and 8720 h (1 year) at both 650 °C and 750 °C. In this way, the microstructural evolution of both steals as a function of tempering time and temperature could be observed.

The specimens prepared for the SEM imaging were usable also for the Vickers hardness measurements. The HV5 measurements were carried out with an Instron 2100B Vickers hardness tester. The measurements were performed before the isothermal tempering, i.e., in the as-delivered state and within the used tempering times. Three measurements were performed over the whole specimen area at a suitable distance from the specimen edge to avoid any edge inaccuracy.

3 RESULTS AND DISCUSSION

A comparison between a decrease in the yield stress (σ_y) and the tensile strength (σ_m) at both tempering temperatures indicates a similarity in the changing of these two properties for both steels.

From **Figures 1** and **2** it can be seen that the effect of tempering at 650 °C on the reduction of σ_m and σ_y is higher for the X20CrMoV121 steel, where σ_y drops by 34 N/mm² and σ_m by 55 N/mm², than for the P91 steel, where σ_y drops by 19 N/mm² and σ_m by 24 N/mm². It is

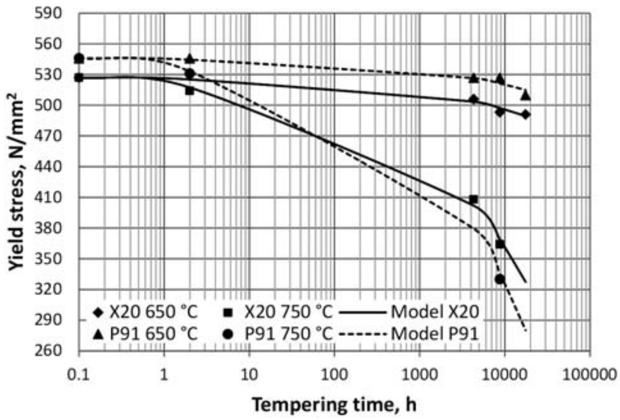

Figure 1: Actual and calculated dependences of the yield stress of the X20CrMoV121 and P91 steels on the tempering time at 650 °C and 750 °C

Slika 1: Dejanska in izračunana odvisnost meje plastičnosti jekel X20CrMoV121 in P91 od časa popuščanja pri 650 °C in 750 °C

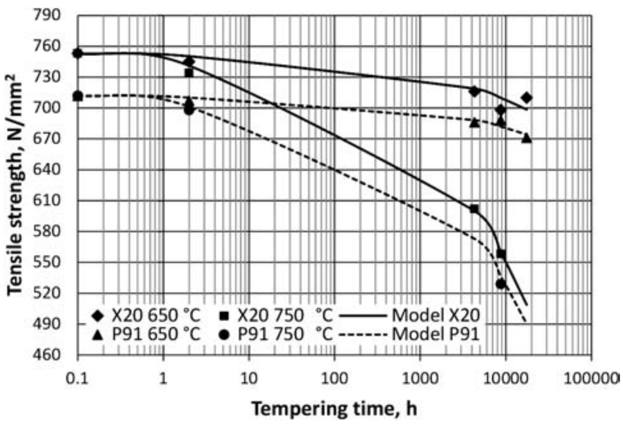

Figure 2: Actual and calculated dependences of the tensile strength of the X20CrMoV121 and P91 steels on the tempering time at 650 °C and 750 °C

Slika 2: Dejanska in izračunana odvisnost raztržne trdnosti jekel X20CrMoV121 in P91 od časa popuščanja pri 650 °C in 750 °C

Table 1: Chemical compositions of the X20CrMoV121 and P91 steels in mass fractions

Tabela 1: Kemična sestava jekel X20CrMoV121 in P91 v masnih deležih

Chemical composition, *w*/%													
Elements	C	Si	Mn	P	S	Cr	Ni	Mo	V	Cu	Nb	Al	N
X20CrMoV121	0.2	0.29	0.52	0.019	0.011	11	0.64	0.94	0.31	0.059	0.024	0.032	0.017
P91	0.1	0.38	0.48	0.012	0.002	7.9	0.26	0.98	0.23	0.14	0.11	0.016	0.064

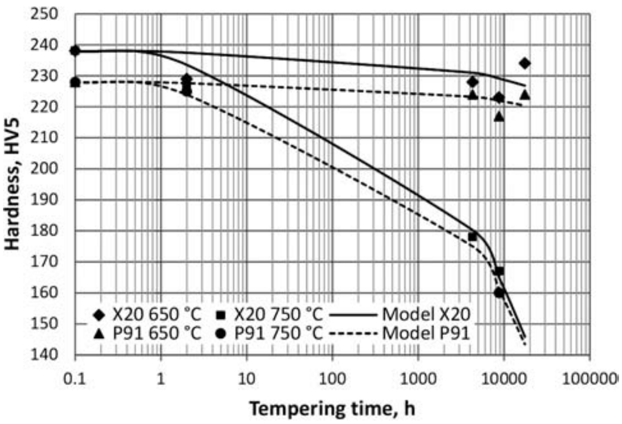

Figure 3: Actual and calculated dependences of the hardness of the X20CrMoV121 and P91 steels on the tempering time at 650 °C and 750 °C

Slika 3: Dejanska in izračunana odvisnost trdote jekel X20CrMoV121 in P91 od časa popuščanja pri 650 °C in 750 °C

also obvious that for both steels the reduction of σ_m is higher than the reduction of σ_y. It should be also pointed out that after a longer tempering time, i.e., 17520 h at the above temperature, the reduction of σ_y is, surprisingly, identical for both steels, i.e., 36 N/mm², and similar behaviour was observed in the reduction of σ_m, i.e., 43 N/mm² for X20CrMoV121 and 41 N/mm² for P91.

On the other hand, the effect of tempering at 750 °C on the reduction of σ_m and σ_y is higher and their mutual correlation is different than in the case of tempering at 650 °C. For X20CrMoV121 σ_y drops by 163 N/mm² and σ_m by 195 N/mm², whereas for P91 σ_y drops by 216 N/mm² and σ_m by 183 N/mm².

The effect of the duration and the temperature of tempering on the hardness of both steels, shown in **Figure 3**, is similar to the effect on the yield stress and the tensile strength. In X20CrMoV121, after 8760 h of tempering at 650 °C, the hardness is reduced by 15 HV, whereas in P91, under the same tempering conditions, the hardness is reduced by 11 HV. At a higher temperature and the same tempering time, i.e., at 750 °C for 8760 h, the hardness reduction in X20CrMoV121 is 71 HV, whereas in P91 this reduction is 68 HV. The reduction of hardness indicates that the size, the amount and the distribution of precipitates have a lower effect on the hardness than on the yield stress of the steels investigated. This could be explained with the fact that the yield stress is more related to deformation hardening than hardness.

There is a clear similarity between the dependences of hardness HV, tensile strength σ_m and yield stress σ_y on the tempering time and the temperature and, for all three properties the general mathematical expression was deduced:

$$y(t) = k_1 - k_2 t^x$$

where $y(t)$ stands for either σ_m, σ_y or HV as a function of tempering time t, k_1 and k_2 are constants and x represents the exponent, which can take the values of 1/2, 1/3, etc., depending on the way the curve obtained from the equation (1) best fits the experimental data of each property.

Experimental vs. theoretical curve fittings are given in **Figures 1, 2** and **3**. They are obtained by using the values for x, k_1 and k_2 given in **Table 2** and applying them in equation (1) for all three properties, both steels and both tempering temperatures. The value of exponent x was appropriated to 1/3, k_1 holds the values for each of the measured properties at the as-delivered state, whereas the k_2 was calculated with the least-square method using the R-project for statistical computing.[13] It should be pointed out that the fitting for the yield stress and the tensile strength is quite good, whereas for the hardness this equation does not give a good fit for a longer tempering time at 650 °C.

Table 2: Parameters for calculating the yield stress, tensile strength and hardness change of the X20CrMoV121 and P91 steels for both tempering temperatures with equation (1)

Tabela 2: Parametri za izračun meje plastičnosti, raztržne trdnosti in trdote za jekli X20CrMoV121 in P91 pri obeh temperaturah popuščanja z enačbo (1)

Properties	Parameters	X20CrMoV121		P91	
		650 °C	750 °C	650 °C	750 °C
σ_y	k_1	527	527	546	546
	k_2	1.444	7.682	1.197	10.231
	x	1/3	1/3	1/3	1/3
σ_m	k_1	753	753	712	712
	k_2	2.096	9.4	1.456	8.539
	x	1/3	1/3	1/3	1/3
HV	k_1	238	238	228	228
	k_2	0.43	3.548	0.292	3.256
	x	1/3	1/3	1/3	1/3

The influence of tempering on the microstructures of both steels is greater after tempering at 750 °C than at

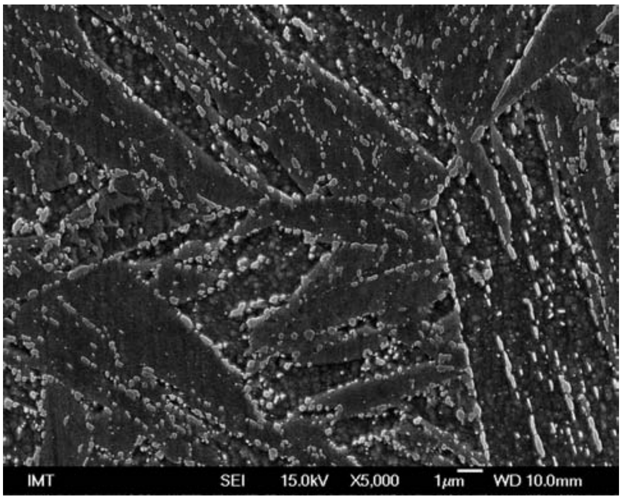

Figure 4: Microstructure of the X20CrMoV121 steel at the initial (as-received) state

Slika 4: Mikrostruktura jekla X20CrMoV121 v začetnem (dobavljenem) stanju

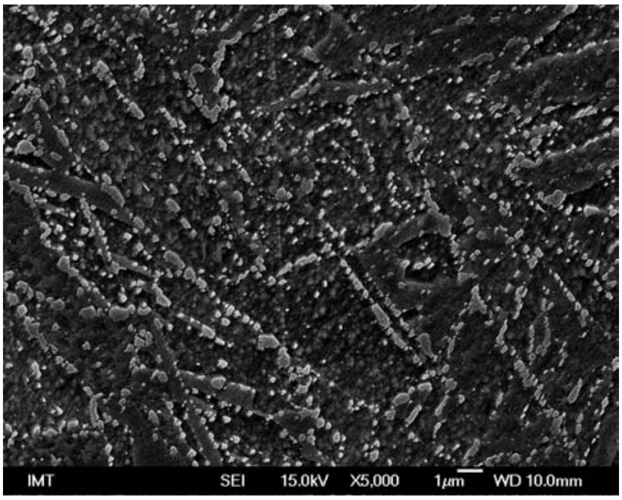

Figure 5: Microstructure of the X20CrMoV121 steel after 8760 h of tempering at 650 °C
Slika 5: Mikrostruktura jekla X20CrMoV121 po 8760 h popuščanja pri 650 °C

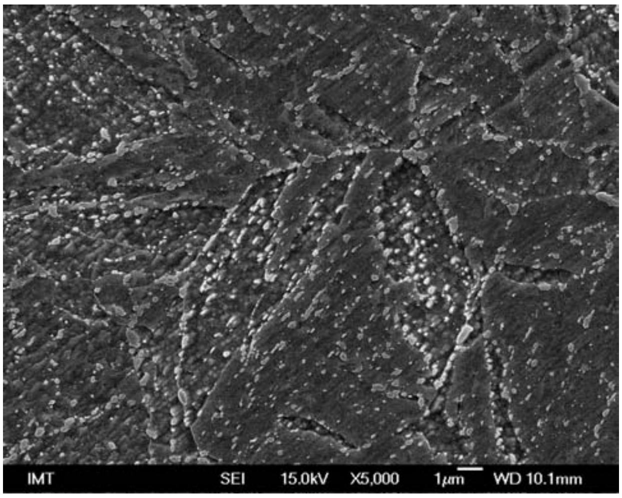

Figure 7: Microstructure of the P91 steel at the initial (as-received) state
Slika 7: Mikrostruktura jekla P91 v začetnem (dobavljenem) stanju

650 °C (**Figures 4** to **9**), because the diffusivity of the carbide-forming elements (Cr, Mo, Fe, V, and Nb), found in the solid solution in the ferrite matrix is temperature dependent, and is higher at higher temperatures.[14–17]

Due to tempering, both the size and the inter-particle spacing of carbide particles increase. In addition, the carbide-particles distribution changes from stringers along the grain and sub-grain boundaries to a uniform structure. Images in **Figures 4** and **7** show the as-delivered-state microstructures of the X20CrMoV121 and P91 steels, respectively. In both cases, the majority of particles are cementite Fe_3C, containing also chromium, or they are chromium carbides $Cr_{23}C_6$, containing also iron and molybdenum.[18,19] The carbide particles are found in the stringers distributed along the grain and sub-grain boundaries of martensite, and there is no difference between the size of the Fe_3C, $M_{23}C_6$ and VC particles. After 8760 h of tempering at 650 °C the precipitates are almost evenly distributed and there is a difference between the size of VC (small white particles) and $M_{23}C_6$, which grow larger in both steels (**Figures 5** and **8**). In addition, the grain and subgrain boundaries of martensite are much less pronounced and some of them have already disappeared.

From **Figures 6** and **9** it is obvious that the tempering at 750 °C for 8760 h causes much greater changes in the microstructures of both steels, where the size of the $M_{23}C_6$ particles and the spacing between them is drastically increased. On the other hand, the number density of these particles has clearly dropped, so the Ostwald ripening effect, where larger particles coarsen at the expense of smaller ones is quite obvious in this case.

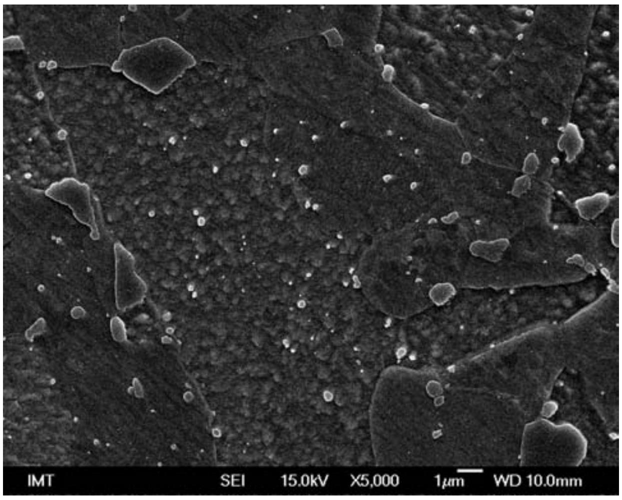

Figure 6: Microstructure of the X20CrMoV121 steel after 8760 h of tempering at 750 °C
Slika 6: Mikrostruktura jekla X20CrMoV121 po 8760 h popuščanja pri 750 °C

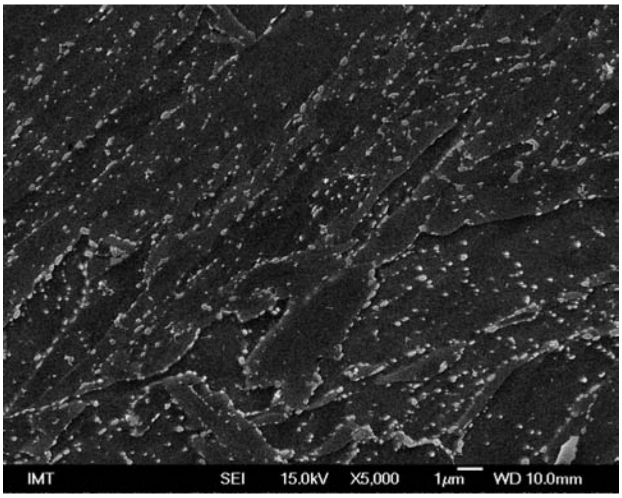

Figure 8: Microstructure of the P91 steel after 8760 h of tempering at 650 °C
Slika 8: Mikrostruktura jekla P91 po 8760 h popuščanja pri 650 °C

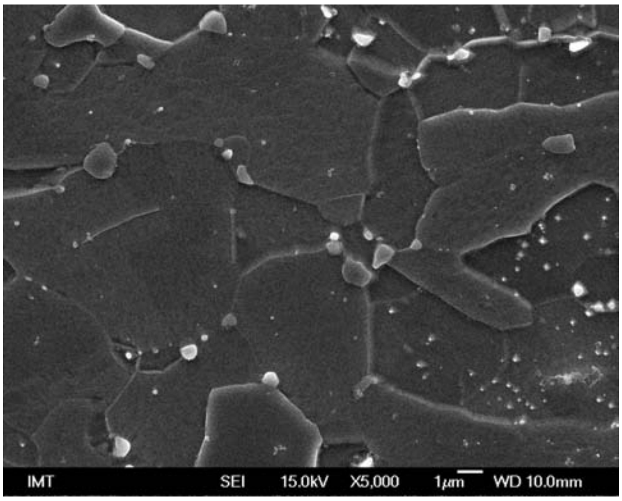

Figure 9: Microstructure of the P91 steel after 8760 h of tempering at 750 °C
Slika 9: Mikrostruktura jekla P91 po 8760 h popuščanja pri 750 °C

Since carbide precipitates represent the most important strengthening mechanism in 9–12% Cr steels, and having in mind the fact that mechanical properties deteriorate as both the size and the inter-particle spacing increase, it is obvious that they are in some kind of mutual correlation, investigated and confirmed by many authors.[14–18]

4 CONCLUSIONS

The effect of the tempering time and the temperature of the creep-resistant martensitic steels, X20CrMoV121 and P91, which differ in chemical composition, on the room-temperature tensile properties and hardness was determined.

According to the results obtained, the following is concluded:

- The effect of tempering at both temperatures separately is the same for both steels investigated.
- When tempering at the temperature of 650 °C, the changes in yield stress σ_y and tensile strength σ_m are relatively small, whereas when tempering at a higher temperature, i.e., 750 °C, the changes are greater for both properties, and for steel X20CrMoV121 σ_y is decreased by 163 N/mm^2 and σ_m by 195 N/mm^2, whereas for steel P91 σ_y is decreased by 216 N/mm^2 and σ_m by 183 N/mm^2 after 8760 h of tempering at 750 °C.
- The dependence of σ_y and σ_m on the tempering time and the temperature is for both steels virtually the same and can be mathematically expressed with the relation: $y(t)=k_1-k_2t^x$ All three parameters, k_1, k_1 and x have different values depending on the tempering conditions, the steel chemical composition, etc.
- Hardness evolution is similar to that of σ_y and σ_m and it could therefore be expressed with the same mathematical relation, however, with different values for all three parameters.
- The effect of microstructural changes, particle size and spacing as well as particle distribution is caused by coarsening the particles. The coarsening rate depends on diffusion, which is greater at higher temperatures.

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