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Relation between macroscopic shrinking and microstructural evolution in 17-4PH steel.

R. Sowa[1], A. Dziedzic[2], S. Arabasz[3], I. dul[4], M. Parlinska-Wojtan[1]

[1]University of Rzeszow, Center for Microelectronics & Nanotechnology, Rzeszow, Poland
[2]University of Rzeszow, Center for Innovation & KNowledge Transfer, Rzeszow, Poland
[3]Labsoft SA, warszawa, Poland
[4]WSK PZL-Rzeszow, Rzeszow, Poland

sowa.roamn89@gmail.com
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The investigated material is a corrosion-resistant, Cu precipitation hardened steel 17-4PH, which shrinks in an uncontrolled way, as a result of the following heat treatment: solution treatment at 1028C for 1 h (condition A), ageing at 540C for 4 h (condition H1025), [1-5]. The aim of this study is to clarify and understand the behavior of the material during the above defined thermal processes based on chemical analysis, microstructure combined with micro-hardness measurement and identification of phase transitions in subsequent stages of heat treatment.

The phase identification was made by X-ray diffraction (XRD) and microstructural imaging was performed using transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM) and scanning electron microscopy (SEM). The chemical analysis was accomplished by energy dispersive X-ray (EDX), and the TEM lamella, were cut by focused ion beam (FIB) technique.

The results were obtained from four samples differing in the applied heat treatment, which is shown in Table 1. Sample 1 is derived from the state of delivery (condition A), sample 2 was again solution treated with the same parameters as before (condition A+A), Sample 3 underwent additionally ageing (condition A+A+H1025), while sample 4 was again solution treatment (condition A+A+H1025+A).

Analysis of the microstructure of the 4 samples shows distinct differences between the states. Figure 1 shows SEM images the cross-sections of the four samples, which were polished and subsequently etched to reveal the microstructure topography. Based on these photographs, it can be clearly seen, that the use of additional heat-treatment operations on samples 1, 2, 3 contributes to significant and irregular grain growth compared to the delivery condition, Figure 1 (a)-(c). Consequently, it is expected that a change of hardness and yield point will occur with a changing grain size. Furthermore, similarities in the microstructure of samples 2, Figure 1(b) and 4, Figure 1(d), such as the presence of lath martensite, in form of parallel stripes and residual austenite – distinct grain boundaries, can be cleary observed. This interpretation is in agreement with the TEM and STEM observations of internal microstructure (images not shown here), where finer grains are visible in sample 1 compared to sample 2. Sample 4 exhibits a large quantity of lath martensite, which contains many dislocations, Figure 1(e).

In all samples, the TEM and the STEM images reveal the presence of, Nb carbides with sizes varying from 50 to 150 nm, confirmed by chemical analysis, Figure 2(a). In the case of sample 3, a large quantity of Cu precipitates, which did not appear in samples 1 and 2 was observed. The Cu content was confirmed by chemical analysis, Figure 2(b). After the solution treatment, these precipitates almost disappeared in sample 4. However, in areas with a high density of dislocations, individual, residual copper precipitates could be identified by EDX. Analysis of the XRD diffractogramms allowed the identification and comparison of phases observed in SEM and TEM photographs, as well as relating the change of lattice parameters to the observed phase transitions and macroscopic shrinking of the material.

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	Condition A 1028°C 1h	Additional Condition A	Aging = 551°C 4h	Condition A 1028°C 1h
Sample 1	Condition A			
Sample 2		Condition A+A		
Sample 3			Condition A+A+H1025	
Sample 4				Condition A+A+1025+A

Table 1.heat treatment procedure applied to the 17-4 PH steel samples

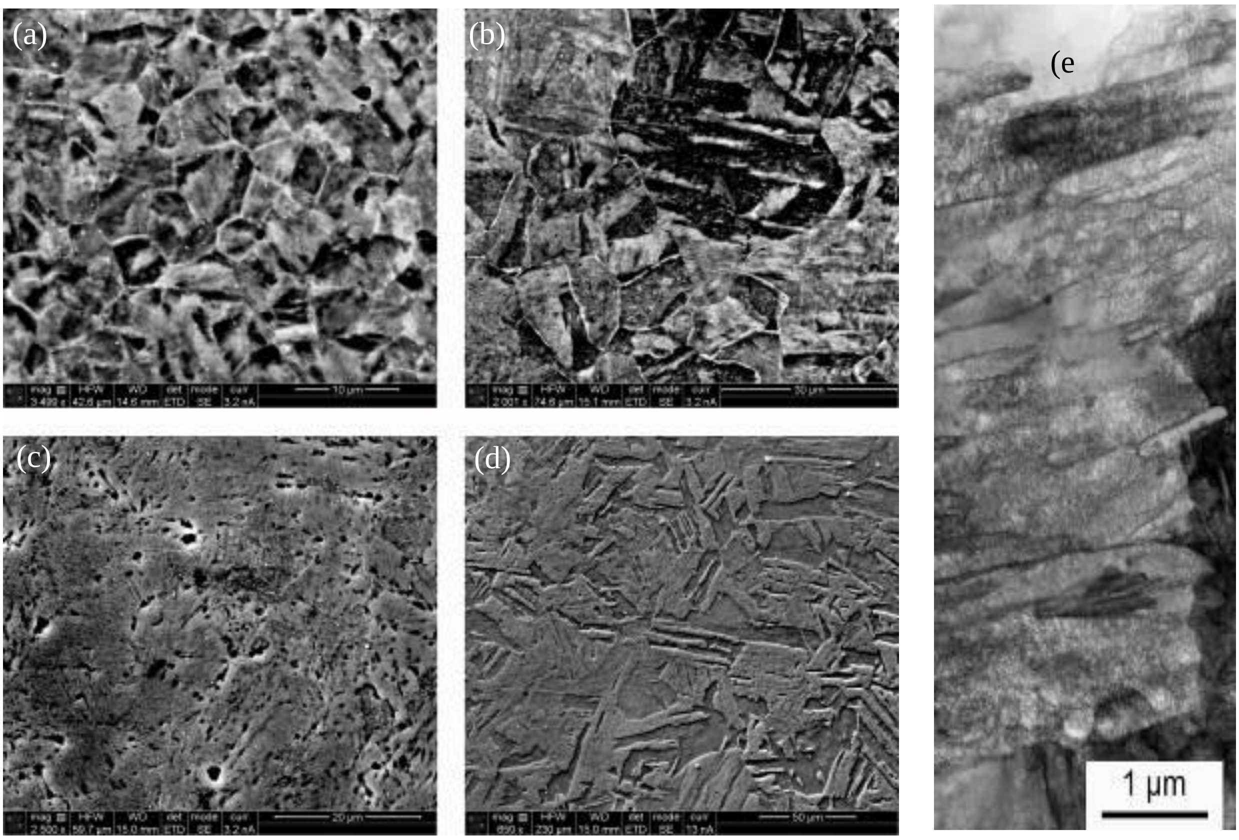

Figure 13. SEM micrographs of polished and chemically etched sample cross-sections illustrating differences between them: (a) Sample 1, condition A; (b) Sample 2, condition A+A; (c) Sample 3, condition A+A+H1025, (d) Sample 4, condition A+A+H1025+A. Please note that the scales on the images are different.

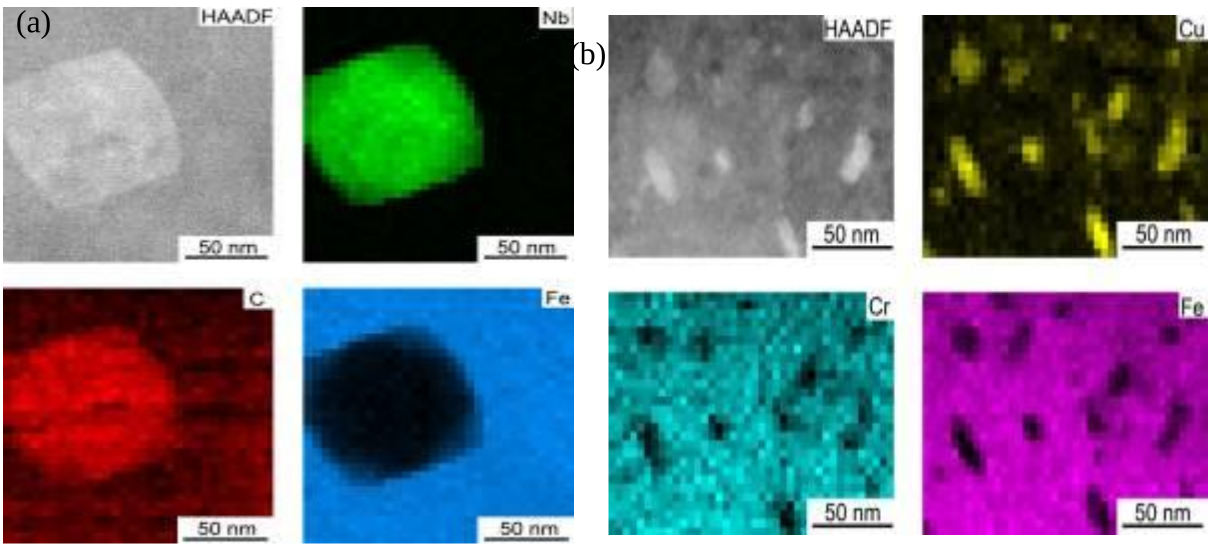

Figure 14. HAADF images and EDX maps of: (a) NbC precipitates present the microstructure of all 17-4PH steel samples; (b) Cu nanoprecipitates observed in the microstructure of sample 3.