TEM investigations of the precipitation kinetics of Mn(Cu)S and AlN in microalloyed steel

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Transmission electron microscopy (TEM) investigations are carried out on a microalloyed steel in order to analyze the precipitation kinetics of MnS and AlN (see also [1, 2]). The results will then be further used for improving thermodynamic calculation algorithms of precipitation kinetics and nucleation processes of precipitates in steel. Its importance will increase in future as a tool for time and cost reduction in steel production.

The specimens with the main alloying elements of the composition (in [wt%]): Mn 0.371, S 0.0059, Cu 0.012, Al 0.14, N 0.0044, C 0.002 and Fe balanced have been heat treated at two different temperatures (950°C and 1050°C), and 5 different annealing times (between 10s and 10.000sec), see also Table 1. The Mn(Cu)S precipitates mostly were spherical (see Fig. 1), so a mean diameter of the particles can be determined. The AlN were needle- or platelike shaped, therefore a length and a width were measured, see Table 1 and Fig. 2.

For TEM investigation, the specimens were prepared as disks with a diameter of 3mm and a thickness of 0.3mm and further electropolished until being transparent for a 200 keV electron probe. The TEM investigations were carried out on a Tecnai F20 FEGTEM. The composition of the precipitates was analysed by using energy dispersive X-ray analysis (EDX), typical EDX spectra of the Mn-S, Mn-Cu-S and Al-N precipitates are given in Fig. 2 and Fig. 3.

It could be shown that the size of both AlN and Mn(Cu)S is changing with temperature and annealing time, details see Tab. 1. Mn(Cu)S particles are formed already after 10sec annealing time at both temperatures, whereas AlN seems to appear as precipitates after 300sec at 950°C and 100sec at 1050°C.

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	950°C				
Time [s]	10	100	300	1.000	10.000
Mn(Cu)S	3-26	20-67	14-28	10-80	8-60
AlN (width)	-	-	56-218	30-67	17-70
AlN (length)	-	-	125-442	80-165	117-330

	1050°C				
Time [s]	10	33	100	1.000	10.000
Mn(Cu)S	18-48	42-68	39-140	56-98	56-90
AlN (width)	-	-	47-67	6-47	43-465
AlN (length)	-	-	437-1678	527-1258	192-1703

Table 1. Particle sizes [nm] of Mn(Cu)S and AlN at different heat treatments.

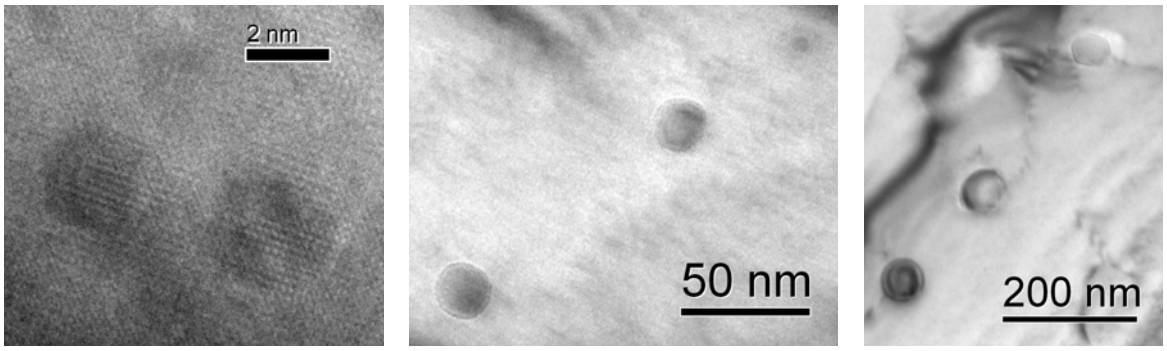

Figure 1. Mn(Cu)S nanoparticles (left) and larger precipitates (center) of heat treatment 950°C 10sec, and heat treatment 1050°C 100sec (right).

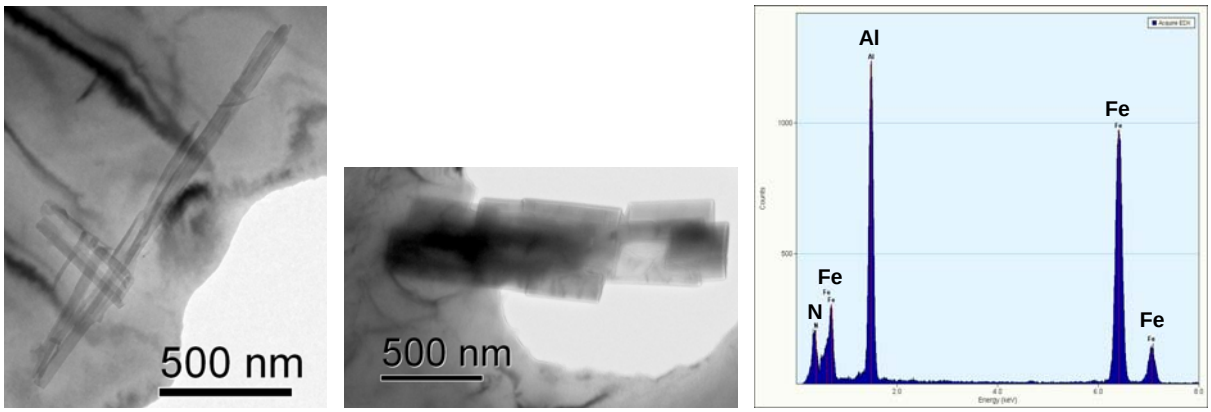

Figure 2. AlN precipitates of heat treatment 1050°C 100sec (left) and 1050°C 10.000sec (center), and typical EDX spectrum of AlN (right).

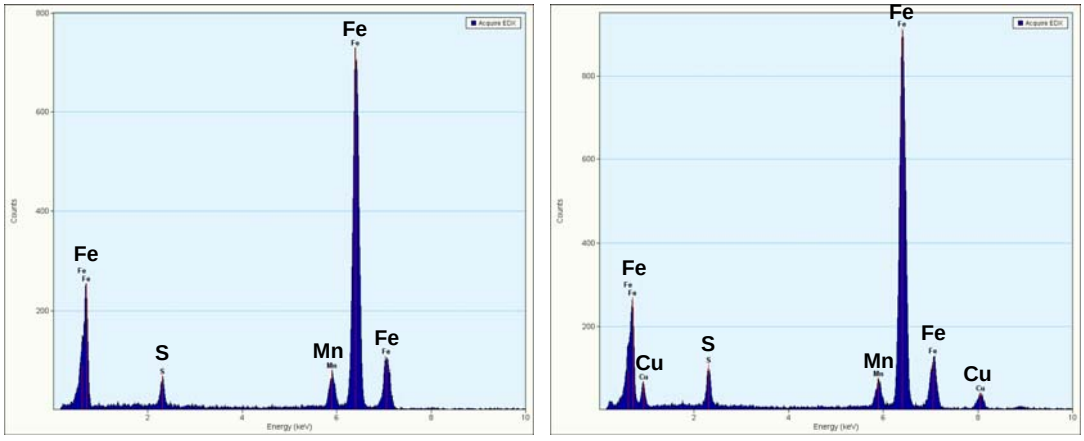

Figure 3. Typical EDX spectra of pure Mn-S (left) and Mn-Cu-S (right).

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