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IT-5-P-2335 WDX-measurement of Ta-, W- and Re-concentration profiles in a Nickel/Superalloy diffusion couple using Lβ-X-ray-lines

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Ni-base superalloys are multicomponent alloys used at temperatures up to about 1100°C. At such high temperatures, diffusion plays the principal role for structural stability and mechanical behaviour. The material under investigation is CMSX-10, which consists of 11 elements (Al, Ti, Co, Cr, Ni-base, Nb, Mo, Hf, **Ta, W, Re**). CMSX-10 is diffusion welded with pure Ni under vacuum at 1050°C, 10 MPa, 1 h, then annealed at 1050°C for 128 days. In order to quantify the diffusion kinetics in such a multicomponent system, the diffusion profiles in Ni/CMSX-10 diffusion couples have to be measured. However, for the key strengthening elements Ta, W and Re this task is not trivial because they are neighbours in the periodic system (atomic numbers 73, 74, 75) and their concentrations are quite small, 1-3 at%. Therefore, X-ray peaks of these elements are small and they overlap. For these reasons, an optimised method is presented.
Measurement of the diffusion profile by EDX-microanalysis in a SEM is not quite reliable because the energy resolution of the detector is too large (127 eV @ 5.9 keV), as can be seen from the overlapping of the M-lines of Ta, W and Re in Figure 1 and the L-Lines in Figure 2, respectively. Thus, WDX-analysis with high energy resolution becomes essential, in our case with the Field Emission Gun Electron Probe Microanalyser (FEG-EPMA) JEOL JXA-8530F, having a resolution of about 15 eV @ 5.9 keV (LIF). However, figure 1 shows, that even now the Ta-Mβ- and W-Mα-lines cannot be separated (ΔE=9 eV) as well as the W-Mβ- and Re-Mα-lines (ΔE= 8 eV).
Energies of the L-lines are in general about 5 times higher than those of the M-lines, thus also the separation of the lines. In Figure 2 it can be seen, that the Lα-peaks of W and Re are isolated, however, the very close and strong Ni-Kβ-line falsifies their background. Therefore, the Lβ-lines are used. The energy differences between TaLβ2 and WLβ1 (ΔE=20 eV) as well as WLβ2 and ReLβ1 (ΔE=50 eV) are large enough to allow a reliable peak deconvolution. To excite the L-lines of Ta, W and Re (E≈10 keV), a 20 kV accelerating voltage is applied. Anyhow, the small Ta-, W- and Re-concentrations give only small peak/background ratios, making necessary a careful background subtraction. The method was checked by measuring the element concentrations in CMSX-10, which gives results very close to the nominal composition. The diffusion profiles were measured 1.8 mm across the interface with a step size of 5 µm. Figure 3 shows the profile scan for Ta-, W-, Re-, Ni and Al. Comparison of the experimental concentration profiles with such modelled by the software DICTRA shows a very close match. Therefore, it is proved that Lβ-lines might be used for the quantitative element analysis in WDX.

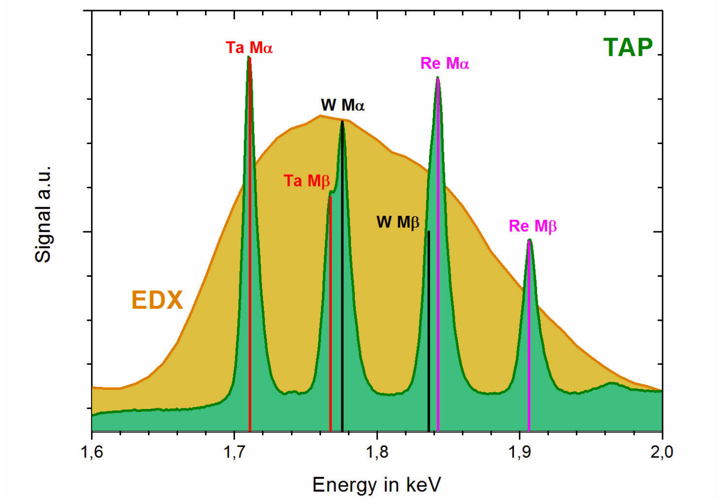

Fig. 1: M-lines of Ta, W and Re in an EDX/WDX-spectrum of CMSX-10

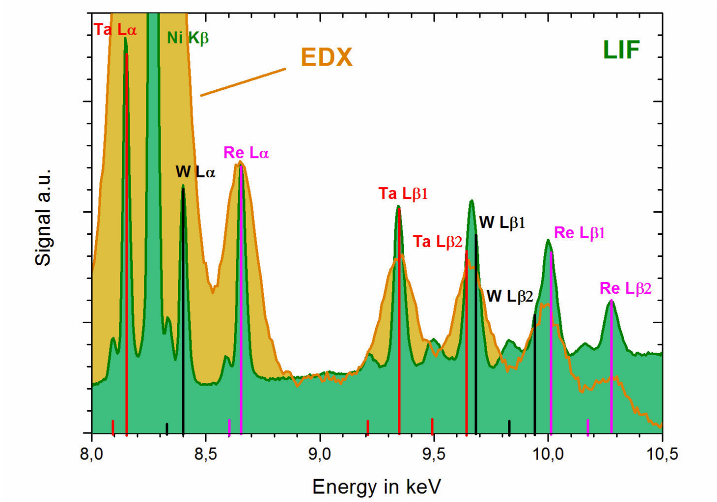

Fig. 2: L-lines of Ta, W and Re in an EDX/WDX-spectrum of CMSX-10

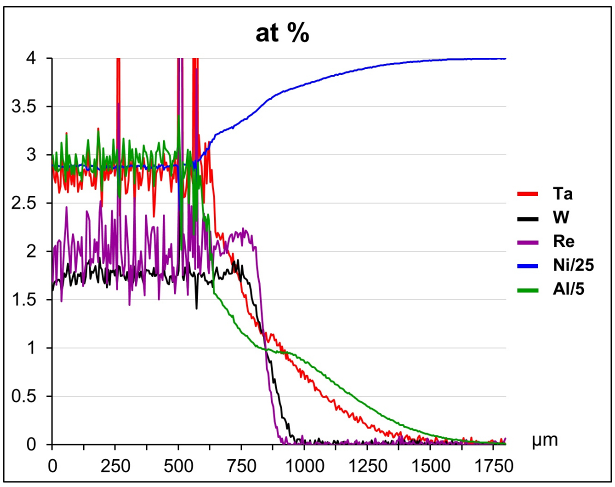

Fig. 3: WDX-Profile scan of Ta, W, Re, Ni and Al in CMSX-10