

# In-situ XRD Studies on the Formation of Expanded Austenite

Degree programme: Master of Science in Engineering | Specialisation: Industrial Technologies

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This master thesis examines the kinetics of expanded austenite formation on AISI 316L by in-situ observation of its X-ray diffraction pattern development during gaseous low temperature nitrocarburization. This novel method allows for collection of detail information about the formation process of the superhard layer with a relatively fine temporal resolution. The influence mechanisms of several parameters relevant to the thermochemical treatment process are investigated.

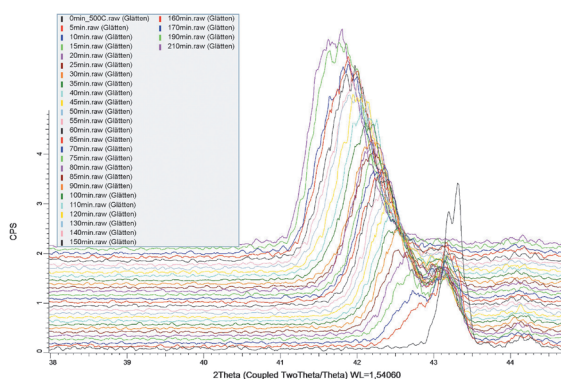
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In-situ XRD analysis during the thermochemical treatment of AISI 316L stainless steel was successfully deployed and delivered useful measurement results: It was possible to obtain information about the formation kinetics of expanded austenite under consideration of several parameters that are relevant to the process of thermochemical treatment. Furthermore, the in-situ XRD pattern development gives insight into type and extent of the ongoing structural changes, such as lattice expansion and lattice defects. The XRD data was supported by metallurgical analysis, microhardness measurements and glow discharge optical emission spectroscopy, revealing the specimen properties after thermochemical treatment. For each experiment, a diffusion coefficient could be determined by measuring the decrease of substrate peak intensity against treatment duration. It turned out that this initial diffusion coefficient does not remain constant during the whole treatment, but shrinks with increasing layer thickness. The effect of surface activation turned out to account for a diffusion coefficient enhancement in the range of one order of magnitude when comparing the treatment of a non-activated surface with the treatment of

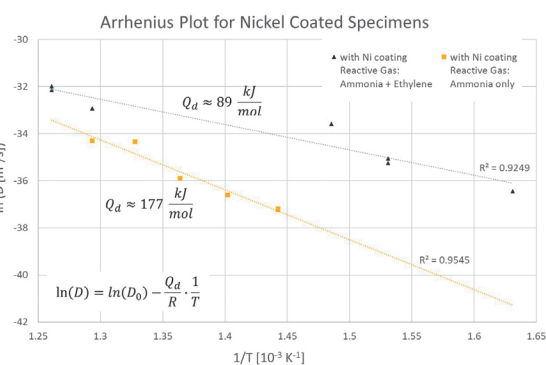
an in-situ HCl depassivated surface. With additional nickel coating – serving as a barrier for repassivation and catalyst for ammonia dissociation – the diffusion was accelerated by two orders of magnitude. Injection of ethylene – as a supplier for carbon atoms – additionally to ammonia showed a clear acceleration of the diffusion process as compared to the treatment with ammonia only. The higher diffusion coefficients observed in-situ could be validated by metallographic layer thickness measurements. The activation energy of interstitial diffusion could be reduced from 177kJ/mol to 89kJ/mol by addition of ethylene. The influence of the microstructural condition of the substrate material before thermochemical treatment was examined. Annealing turned out to decelerate the interstitial diffusion process: the activation energy increases from 140kJ/mol for cold worked AISI 316L to 201kJ/mol for the annealed version. A higher amount of lattice defects in the cold worked matrix observed by diffraction pattern comparison is most probably responsible for faster diffusion in those samples. This finding could be validated by comparison of S-phase layer thickness values.



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Waterfall view of a diffraction pattern development



Arrhenius type plot for the combined diffusion coefficients of nitrogen and carbon in nickel coated AISI 316L