Bern University of Applied Sciences
Engineering and Information Technology

Surface characterization of thermally processed, stainless steels by GD-OES

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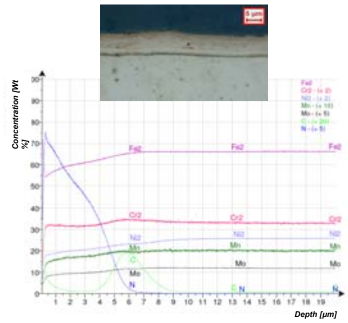
Abstract :

Austenitic stainless steel offers good chemical resistance in various media and atmospheres. However it is quite soft and its rather low wear resistance limits often the desired function. The object of this work is to contribute to a novel low temperature nitriding process in order to generate a wear-resistant surface layer on austenitic stainless steel. These novel processes lead to the formation of a interstitial solution of nitrogen which induces a strong lattice distortion of the austenite which in turn leads to a significant increase in hardness. The analysis of the hardened surfaces was done by means of GD-OES to have an insight in the depth profiles of the carbon- and nitrogen concentrations.

Novel low temperature nitriding process in order to generate a wear-resistant surface layer on austenitic stainless steel:

Stainless steel with an austenitic crystalline structure offers good resistance to stress-corrosion in various media. Its rather low Vickers-hardness of about 200HV and wear resistance often limits its application.
Many well-known surface hardening processes such as case hardening, gas nitriding, plasma nitriding, nitro-carburization etc. may improve the wear resistance of steels by forming a hard surface layer. However, two problematic features are encountered by using these hardening processes for austenitic stainless steel:

- The chemical resistance is considerably reduced by precipitation of chromium carbides and nitrides at the processing temperatures of more than 500°C
- The passivation layer, responsible for the chemical resistance, hinders the nitrogen or carbon diffusion substantially or even blocks it completely



Different new hardening processes aim to induce a hard and wear resistant surface having a Vickers hardness of more than 1000HV whilst retaining the excellent corrosion resistance.
These novel processes do not lead to the formation of nitrides, as do the classical ones, but to an interstitial solution of nitrogen ("expanded austenite" or "S-phase“) in the outer zone. The interstitial nitrogen atoms induce a strong lattice distortion of the austenite which in turn leads to a significant increase in hardness. The same effect can be obtained by carbon.

For surface characterization of thermally processed stainless steels, quantitative depth profile analysis by GD-OES is a well established, reliable and fast method to determine enrichments or reduction of alloying elements. In order to analyse in detail the depth profiles of the carbon- and nitrogen concentrations in this nitriding process , GD-OES experiments were carried out. The sputtering time for each measurement was five minutes. This short measurement time allows high sample throughput and/or measurement repetition under various process conditions. However, the recalibration of the measured intensity-time curve for quantification, usually takes about one hour.

High nitrogen austenitic stainless steel for micro-technical applications:

In the production process for micro-mechanical components the material passes through several annealing processes. Depending on the annealing parameters (temperature, time, atmosphere) there may appear undesired modifications concerning the chemical composition of the outer zone, which can in turn influence the crystalline structure and therefore the mechanical properties.

Vacuum annealing of the chromium-molybdenum-manganese steel under investigation led to a loss of nitrogen, carbon and manganese in the near-surface zone, and hence to the formation of a ferritic and therefore magnetic edge zone.
However, non-magnetic behaviour is mandatory for the micro-mechanical components in question; the annealing process must therefore be adapted to avoid alterations of the chemical composition which may lead to a shift of the alloy position in the Schaeffler-diagram from the austenitic to the ferritic region.

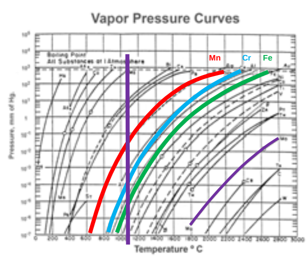
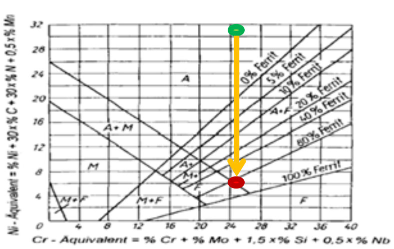
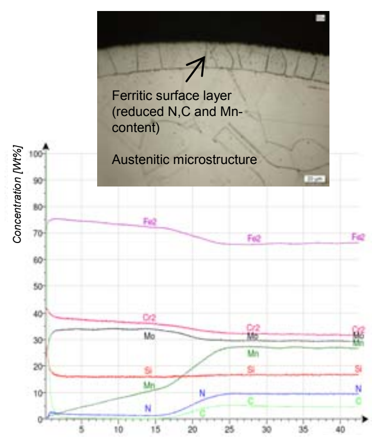


Fig2: Vapor pressure curves for several elements depending on the temperature.



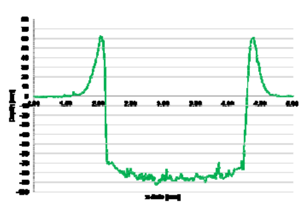
By means of the Schaeffler-diagram and the quantification of the alloying elements Mn, C and N by GD-OES, the modification from the austenitic to the ferritic zone in the near surface region can be calculated.

Depending on the temperature for the annealing process, the alloying elements have different vapour pressures. For the annealing temperature of about 1080°C, manganese has an about 100 times higher vapour pressure than chromium. During the annealing process, manganese evaporates hence at the surface. This leads to a diffusion driven migration of manganese from to the near surface region. Together with the decrease of carbon and nitrogen in this area, the variation of the chemical composition leads to a change of the crystalline structure and hence to different mechanical and magnetic properties. The outer zone of the sample gets magnetic.



GD-OES depth profile of the sample after the annealing process. The decrease of manganese, nitrogen and carbon in the near surface region leads to a change in the microstructure. This ferritic surface layer of about 25 µm thickness, as shown in the micrograph, has a magnetic behaviour.

The samples to be analysed in this project consisted of narrow bands (3 mm x 180 µm) of austenitic steel . We have developed a sample adapter specifically for this particular sample type. It consists of an aluminium work piece, with a custom made groove for the sample and housing for the o-ring, allowing a perfect sealing of the discharge chamber. For the analysis we used the 2.5 mm inner diameter anode, allowing to sputter the sample surface only, i.e. without interference by the adapter. With this adapter, easy measurement repetition can be done .



The crater profile shows the sputtering induced roughness, issue of the distinct crystallinity of the sample.

Presented at : Anwendertreffen Glimmentladung, IFW Dresden, D
November 23./24.11.2011