ARE X

Retained Austenite: Fast and Reliable Solution for Quality Assurance and Quality Control Practices in Heat Treatments

Introduction

Austenite is a very useful structural constituent of advanced high-strength steels. Its ability for strengthening in many different ways offers possibilities to obtain a unique range of mechanical and technological properties. An especially important feature of austenite phase is its ability to transform to martensite or to form twinned microstructures during straining. The first one is utilized in low-alloyed multiphase steels containing from 5 to 15% volume amount of retained austenite. Instead, twinning is a main hardening mechanism in fully austenitic high-manganese alloys.

Physical properties depend on different phase amount, if phase transformation occurs also these physical properties change. Austenite to martensite transformation involves a 4-5% volume increase that can induces strain hardening leading to outstanding combination of strength and ductility (i.e., TRIP effect). Instead, it can also affect the dimensional stability leading to crack initiation.

High content of retained austenite can result in lower elastic limits, reduced hardness, lower high cycle fatigue life and dimensional instability. Instead, low content of retained austenite can result in poor fracture toughness and reduced low cycle fatigue and rolling contact fatigue life.

The amount of retained austenite and its carbon content (besides chemical composition) are highly dependent on a processing.

XRD has long been used to measure the amount of retained austenite in steel. The standard method (ASTM E-975-03) employs ratios of integrated intensities of the austenite and martensite or ferrite phases. The accuracy of the retained austenite determination is also challenged at very low austenite contents (~1 weight %).

Usually retained austenite determination can be better performed by XRD instead of optical microscopy (OM) because it is user-independent. By OM method underestimation is a frequent issue.

Summary

X-ray diffraction (XRD) is a non-destructive analytical technique that can provide reliable solutions to many issues related to material science and industrial applications as quality assurance and quality control practices. XRD applied to retained austenite determination in Heat Treatment is a reliable solution to achieve the final goal of optimizing the manufacturing process and its cost.

Product Specifications

ARE X is a θ–θ diffractometer dedicated to retained austenite determination. Its high-count statistic involves an error on the single measurement is in the 0.02-0.03% range.

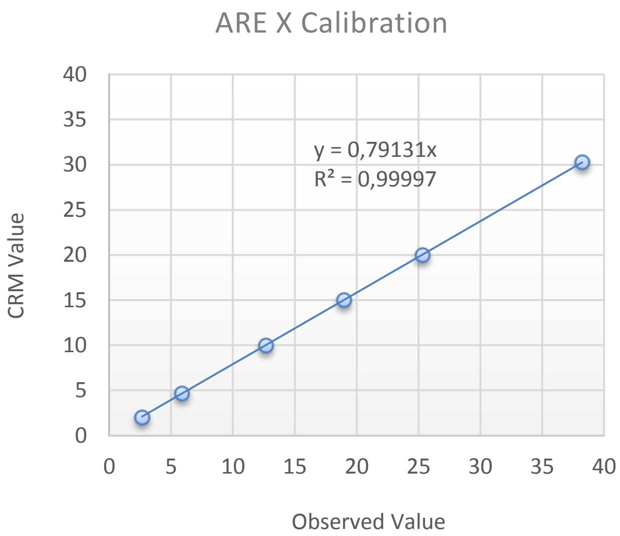
ARE X reproducibility is in the 0.10-0.60% range on three measurements.

ARE X accuracy is in the 0.05-0.50% and it is guaranteed by CRM calibration.

The volume percent of retained austenite in steel is determined by comparing the integrated XRD intensity of ferrite and austenite phases with theoretical intensities as in the following formula:

$$V_\gamma = \frac{1}{q}\sum_{j=1}^{q}\frac{I_{\gamma j}}{R_{\gamma j}}\left(\frac{1}{p}\sum_{i=1}^{p}\frac{I_{\alpha i}}{R_{\alpha i}}+\frac{1}{q}\sum_{j=1}^{q}\frac{I_{\gamma j}}{R_{\gamma j}}\right)$$

Where
V_γ is the volume fraction of austenite,
q is the number of austenite reflections,
p is the number of ferrite reflections,
I is the integrated intensity and
R is a theoretical parameter



Hardware control and data analysis are managed by AUSTENITE software in a user-friendly way (Fig.1). At the end of each measurement, the volume % value of retained austenite is immediately displayed as information about compliance with ASTM E 975-03 (“Standard Practice for X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation").

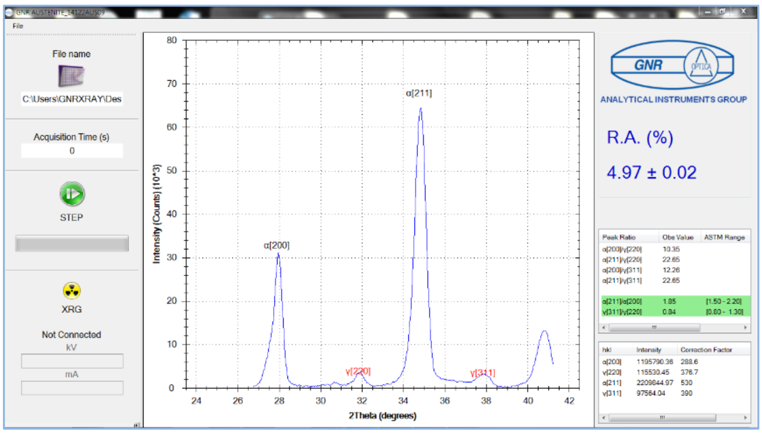


Fig. 1. Austenite Software

Sample Preparation and Measurement Parameters

ARE X allows to analyze both specimens and real samples.

The analyzed samples are specimens of heat-treated UNI 18NiCrMo5 produced by a manufacturing process involving Carburizing, Hardening, Tempering and Blasting steps (CHTB). The aim is to monitor the content of retained austenite as a function of hardening depth, direction of measuring (sample homogeneity) and Cryogenic-Hardening (CH) additional process step.

Very useful advice on sample preparation can be found in E 975-03 and SAE 453.

ARE X Instrumental Setup

Source	Fine Focus Mo
Voltage [kV]	50
Current [mA]	20
Filter (mm)	0.07, Zr
collimator[Ø mm]:	1.0
Detector type	LPSD
Angular range 2θ [°]	26-40
Acquisition time [s]:	60

Results

An example of Quality Assurance application is reported in figure 2 where retained austenite content referring to manufacturing process involving Carburizing, Hardening, Tempering and Blasting, (CHTB). The process has been controlled at different operative conditions by analyzing specimen representative of the overall process. the specimens have been produced by four different process:

1. CHTB with hardening depth of 2.5 mm
2. CHTB with hardening depth of 2.5 mm followed by CH step
3. CHTB with hardening depth of 2.0 mm
4. CHTB with hardening depth of 2.0 mm followed by CH step

Each specimen has been analyzed on the surface and 0.3 mm below the surface and each measurement has been repeated three time. Moreover, the specimens have been analyzed in two direction 90 degrees apart from each other ("_90" and "_0" labels in figure 2) for a total 48 measurements.

Therefore, the characterization of the different processes takes a measuring time of 48 minutes only (60 s for each measurement).

It has been observed, as expected, that the CH additional step drastically lower the content of retained austenite.
Moreover the results show that the hardening process of 2mm lead to a less homogeneous surface than 0.3 mm below as a function of measuring direction in CTHB and CHTB + CH respectively. This difference is more evident in absence of CH step.
Instead, the hardening process of 2.5 mm leads to specimens that are more homogeneous. Retained austenite amount results does not change significantly as a function of depth in CTHB and CHTB + CH respectively.
These results show as ARE X could be the XRD solution dedicated to Quality Assurance and Quality Control practices involved in Heat Treatment manufacturing.

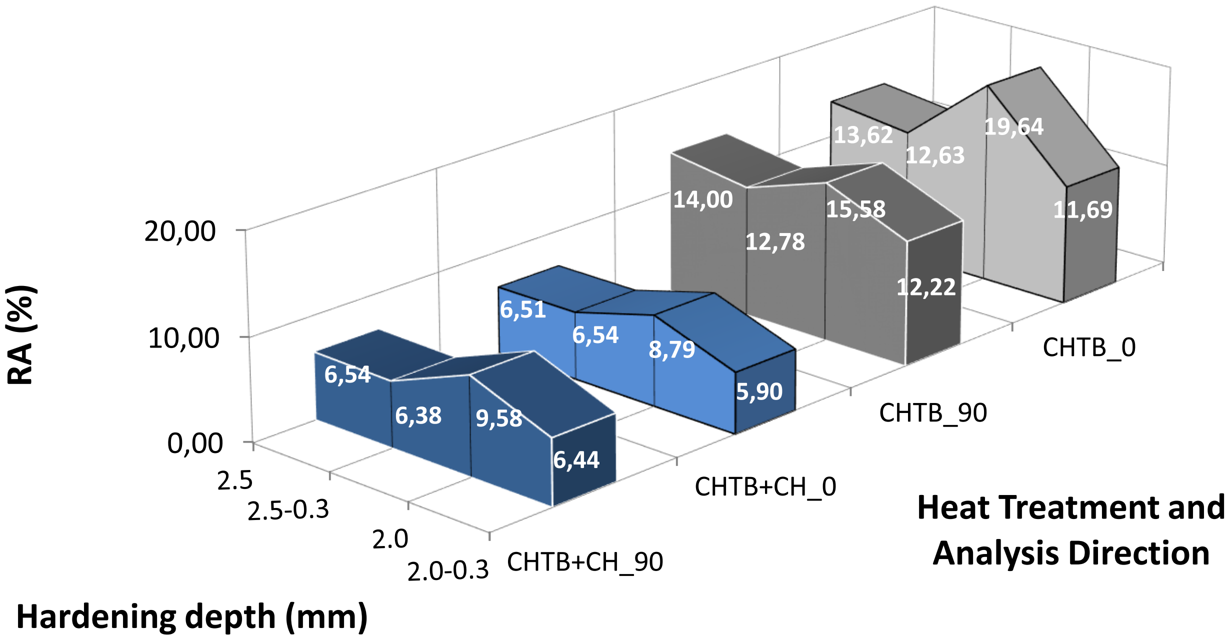


Fig. 2 Retained austenite measurements on of heat-treated UNI 18NiCrMo5 specimens produced by a manufacturing process involving Carburizing, Hardening, Tempering and Blasting (CHTB) and Cryogenic-Hardening (CH) additional step.

Conclusions

All materials with a sufficient degree of crystallinity can be analyzed by X-ray diffraction (XRD) providing useful information about quantitative phase analysis (QPA) and crystal structure.
Retained austenite can be successfully determined by XRD in a non-destructive way and in compliance with standard practice ASTM 975-03. ARE X is a fast and reliable XRD solution to Heat Treatments manufacturing issues as Quality Control and Quality Assurance practices.