ANALYTICAL INSTRUMENTS GROUP

AreX

Retained Austenite Analyzer

www.gnr.it

About us

G.N.R. S.r.l., thanks to its 30 years of experience, is a worldwide market manufacturer of advanced analytical instruments, developing procedures of analysis for various applications, supplying the corresponding laboratory equipment and providing consulting and Customer support worldwide, through its well established sales and post-sale network.

G.N.R. S.r.l. projects and manufactures Optical Emission Spectrometers (OES) and Rotating Disc Electrode Optical Emission Spectrometers (RDE-OES) for the measurement of elemental composition of metal alloys and the analysis of contaminants, additives and wear metals in oils and lubricants, coolants and hydraulic fluids.

G.N.R. S.r.l. designs and produces X-Ray Diffractometers (XRD) and X-Ray Fluorescence Spectrometers (XRF) for the study of structure and elemental composition of materials for both scientific and industrial applications.

GNR Head Office and Production Site is located in Agrate Conturbia (Novara), near Lago Maggiore; 20 minutes from MALPENSA Airport.

Certified Company

Highest quality in our products and services is a core value for GNR.
Full commitment is dedicated to support our quality system in the overall process and continuous improvement is fundamental to guarantee GNR compliance to the internationally accepted quality management standard ISO 9001.

ZERTIFIKAT ◆ CERTIFICATE ◆ 認証 ◆ СЕРТИФИКАТ ◆ CERTIFICADO ◆ CERTIFICAT

CERTIFICATO

Nr 50 100 4258 - Rev. 05

Si attesta che / This is to certify that

IL SISTEMA QUALITÀ DI
THE QUALITY SYSTEM OF

G.N.R. S.r.l.

SEDE LEGALE E OPERATIVA:
REGISTERED OFFICE AND OPERATIONAL SITE:

VIA TORINO 7
I-28010 AGRATE CONTURBIA (NO)

È CONFORME AI REQUISITI DELLA NORMA
HAS BEEN FOUND TO COMPLY WITH THE REQUIREMENTS OF

UNI EN ISO 9001:2008

QUESTO CERTIFICATO È VALIDO PER IL SEGUENTE CAMPO DI APPLICAZIONE
THIS CERTIFICATE IS VALID FOR THE FOLLOWING SCOPE

Progettazione, fabbricazione, assistenza e messa in esercizio di spettometri ad emissione ottica e strumentazione a raggi X per la caratterizzazione dei materiali. Commercializzazione di prodotti associati ed affini (IAF 19, 29)

Design, manufacture, service and start-up of optical emission spectometers and x-ray instruments for materials characterization. Trade of complementary and similar product (IAF 19, 29)

Per l'Organismo di Certificazione
For the Certification Body
TÜV Italia S.r.l.

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G.N.R. periodically organizes at its facility courses and training for technicians and agents as well as seminars and demontrations.

Thanks to an extensive network of agents GNR provides technical support and delivers spare parts worldwide.

In relation to the process of continuous development, GNR reserves the right to change specifications of the instruments without previous notice at any time; the real specifications will always be those shown in the final order confirmation.

GNR Analytical Instrument offers X-Ray Diffraction instruments for measuring retained austenite content and residual stress state.

Austenite is a useful structural constituent of advanced high-strength steels. Its ability to strengthen offers possibility to obtain a unique range of mechanical and technological properties. An important feature of austenite phase is its ability to transform into martensite or into form twinned microstructures during straining.

Accurate measurement of the retained austenite content is important in the development and control of heat treatment process in steel industry.

GNR is proud to offer a unique and easy to use instrument able to measure retained austenite in compliance with **ASTM E 975-03**.

GNR AreX system is a fixed-angular range X-Ray Diffractometer equipped with the most modern technical features, which grant accuracy, precision, safety and easiness of use, specifically designed for quantitative determination of retained austenite.

GNR AreX system is equipped with the following main components:

- Main Unit
- X-Ray Source
- Linear position sensitive detector
- USB Camera
- Software

Thanks to **GNR AreX** innovative concept, retained austenite volume percentage can be measured in few minutes, just positioning the sample and pressing start button.

AreX Features

- Compliant with ASTM E 975-03
- Fast Measurement (< 3 minutes)
- 3000 W X-Ray Generator
- Mo X-Ray Tube with Monocapillary Collimation
- Zirconium K_β filter
- Fast Detector
- Sample Holder (110 x 150 mm)
- Custom Solutions available

Main Unit

GNR AreX Main Unit contains the following components:

- Power Supply
- HV Generator
- Electronic control unit

X-Ray Source

GNR AreX Unit is equipped with a Mo X-Ray Tube. According to the test carried out with different anodes, the Mo source has been established as the optimal one because it allows to detect a large number of diffraction peaks, minimizing texture effects and because it has the best intensity response in comparison to other sources.

Linear position sensitive detector

GNR AreX is equipped with a Multi Strip Detector. GNR adopts DECTRIS MYTHEN X-Ray Detector.

MYTHEN, linear silicon strip detector, based on single photon counting technology, provides noise-free performance, high intensity measurement and fast data acquisition.

The high efficient 1-dimensional multi strip detector simultaneously captures a large angular range and reduces measurement time from hours into minutes.

- Mythen can decrease measurement time significantly down in comparison with a scintillator detector without affecting data quality like intensity, resolution and peak shape.
- Compact size, air cooled (no gas, water o liquid nitrogen needed) and maintenance-free detector.
- Fluorescence background suppression by setting an appropriate energy threshold

MYTHEN2 R	1D
Number or Strips	640
Sensor thickness [µm]	450
Strip width [µm]	50
Strip length [mm]	8
Dynamic range [bit]	24
Energy range [keV]	5-40
Max Couting Rate [counts/s/strip]	$1x10^6$
Frame rate [Hz]	25
Point-spread function [strip]	1
Cooling	Air
Dimensions [WHD mm]	38x62x22
Module Weight [g]	100

USB Video Camera

A professional USB video camera with a resolution of 12 megapixels is mounted inside of **GNR AreX** system and displays the measurement area on the sample surface.

About Retained Austenite

Hardening of steels requires heating to an austenitic phase and quenching to room temperature to produce a hard martensitic phase.

Austenite is a face centered cubic (FCC) phase present in steel at high temperature. Upon cooling, most of the steel is transformed into ferrite – a body centered cubic (BCC) phase – or into martensite – a body central tetragonal (BCT) phase. Depending on the rate of the cooling, some percentage of the steel (typically 0-40%) remains as austenite. Hence, the term "retained austenite".

The volume of the austenitic unit cell is greater than either the martensitic or the ferritic unit cells. The retained austenite remained after heat-treating can further transform during the service life of the product into other phases, involving dimensions changes.

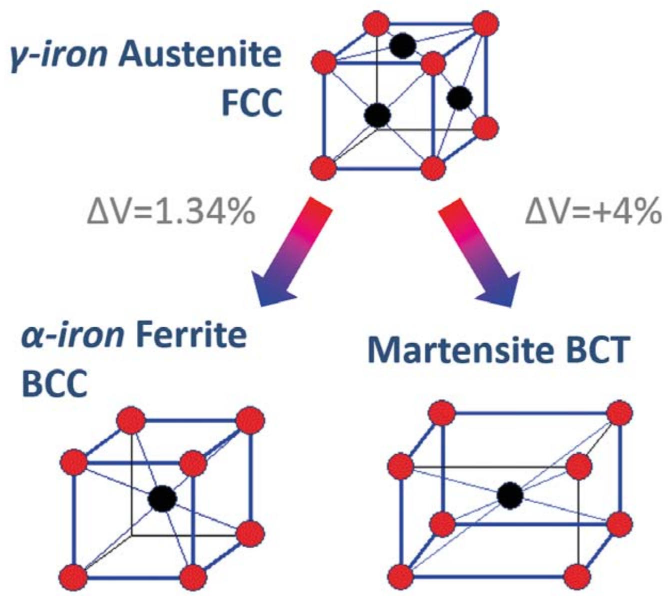
Austenite is a very useful structural constituent of advanced high-strength steels. Its ability to strengthen in many different ways offers possibilities to obtain a unique range of mechanical and technological properties.

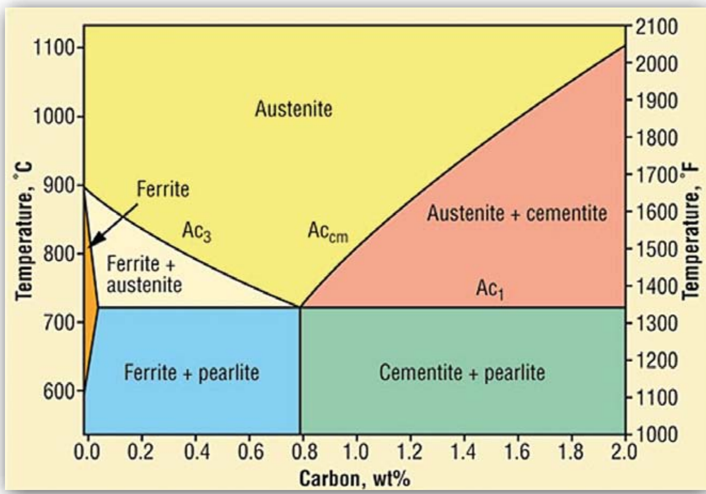
An important feature of austenite phase is its ability to transform into martensite or into form twinned microstructures during straining.

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

In addition, high content of retained austenite can result in lower elastic limits, reduced hardness, lower high cycle fatigue life and lead to dimensional instability. Low content of retained austenite can result in poor fracture toughness and reduced low cycle fatigue and rolling contact fatigue life.





Retained Austenite Analysis

X-Ray Diffraction, providing useful quantitative information about phase content, can be used to analyze all materials with a sufficient degree of crystallinity. This feature is used in retained austenite determination.

Quantitative determination of retained austenite content in heat-treated steels by X-Ray Diffraction has provided a reliable means of controlling production process and ensuring quality.

X-Ray Diffraction is considered to be the most accurate method of determining the amount of retained austenite in steels.

Using “ASTM E 975-03 Standard Practice for X-ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation” along with the AreX instrument, retained austenite content can be easily monitored and controlled.

Austenite, due to its structural difference from other phases in steel, produces a diffraction peak at different angles than ferrite and martensite. The amount of a phase is proportional to the integrated intensity of its diffraction peak.

The amount of retained austenite can be correlated to the ratio of the integrated intensity of the austenite peaks to the integrated intensity of peaks associated with the other phases.

To calculate the volume concentration of retained austenite up to seven (7) diffraction peaks can be collected by **GNR AreX**, three (3) for ferrite/martensite phase and four (4) for the austenite phase.

A comparison of the intensities of the four peaks yields the volume percent concentration of retained austenite in the sample.

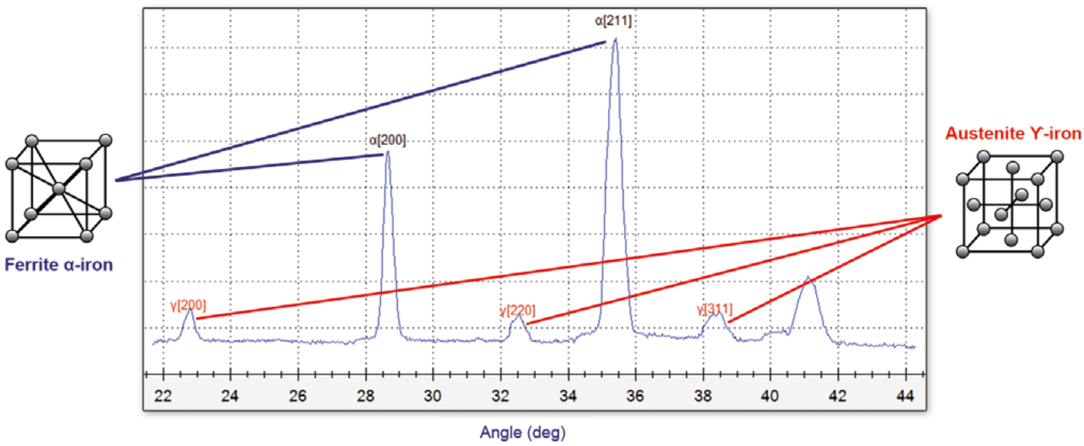
The integrated intensities of the austenite and the ferrite diffraction peaks are measured on the **GNR AreX** diffractometer, providing four austenite/ferrite peak intensity ratios.

The use of multiple diffraction peaks minimizes the effects of preferred orientation and allows interference from carbides to be detected.

Austenite and Ferrite ≠ **Phases** ⇔ ≠ **XRD peak positions**

XRD **Peak Integrated Intensity** (I_i) ∝ Phase$_i$ **Volume Fraction**

Austenite Volume Fraction **RA [%]** $\propto \frac{I_\gamma}{I_\gamma + I_\alpha}$

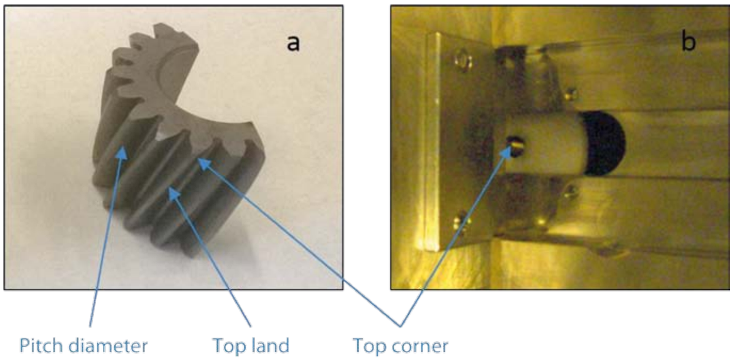


Retained Austenite in Gear Manufacturing

In this specific case, Retained Austenite amount has been determined by using the dedicated diffractometer **GNR AreX** compliant with the standard practice **ASTM E 975-03**.

Retained Austenite (RA %) has been evaluated in three different points in two different samples ("Big Gear" and "Small Gear").

1. Pitch Diameter
2. Top Land
3. Top Corner



Powertrain samples and measured points (a) and pinion sample mounted on teflon support to measure tip corner region (b) taken by inner USB camera.

AreX Instrumental Set Up	
Source	Fine Focus Mo
Voltage [kV]	50
Current [mA]	20
Filter (mm)	0.07, Zr
Collimator [Ø mm]	0,7
Detector type	LPSD
Angular range 2Θ [°]	22-44
Acquisition time [s]	60-120

In order to better perform measurements at points 2 and 3 sample cutting has been necessary. Moreover, in order to measure the top corner region, a teflon support has been used in order to collect X-Rays scattering only from this sample region. In order to measure top corner the best solution is to realize a suitable mask to ease sample positioning.

Each measurement has been repeated three times (M1-M3 in tab.1) in order to calculate average and standard deviation values.

SAMPLE	Measuring Point	RA %				
		M1	M2	M3	Average	SD
Big Gear	Pitch Diameter	15.07	14.21	14.28	14.52	0.39
	Top Land	23.21	23.75	23.28	23.41	0.24
	Top Corner	26.04	25.81	26.27	26.04	0.19
Small Gear	Pitch Diameter	13.45	14.49	13.85	13.93	0.43
	Top Land	20.13	20.84	19.94	20.30	0.39
	Top Corner	24.69	25.15	24.82	24.89	0.19

Tab.1 - Measurement Results

Algorithm error (on single measurement) is in the 0.05 - 0.10 range while **GNR AreX** precision is in the 0.10 - 0.60 range on three measurements.

Samples of Manufacturing Process

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.

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

AreX Instrumental Set Up	
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 [°]	22-44
Acquisition time [s]	60

The specimens have been produced by four different process:

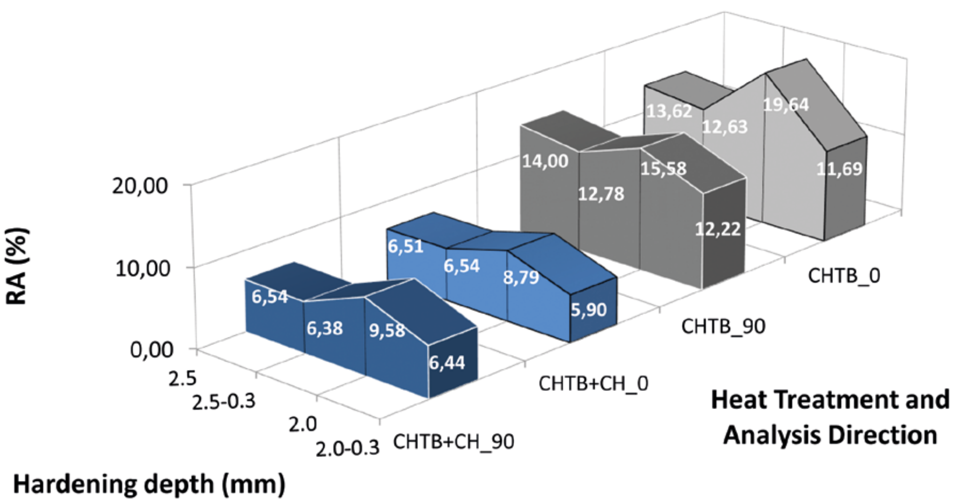
1. CHTB with hardening depth of 2.5 mm
2. CHTB with hardening depth of 2.5 mm followed by CH step
3. CHTB with hardening depth of 2.0 mm
4. CHTB with hardening depth of 2.0 mm followed by CH step

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 lowers the content of retained austenite. Moreover the results show that 2 mm hardening process leads to a less depth homogenity. This difference is more evident in absence of CH step.

The 2.5 mm hardening process leads to specimens that are more homogeneous: retained austenite amount does not change significantly as a function of depth in CTHB and CHTB + CH respectively.

These results show how **GNR AreX** could be the XRD solution dedicated to Quality Assurance and Quality Control practices involved in Heat Treatment Manufacturing.

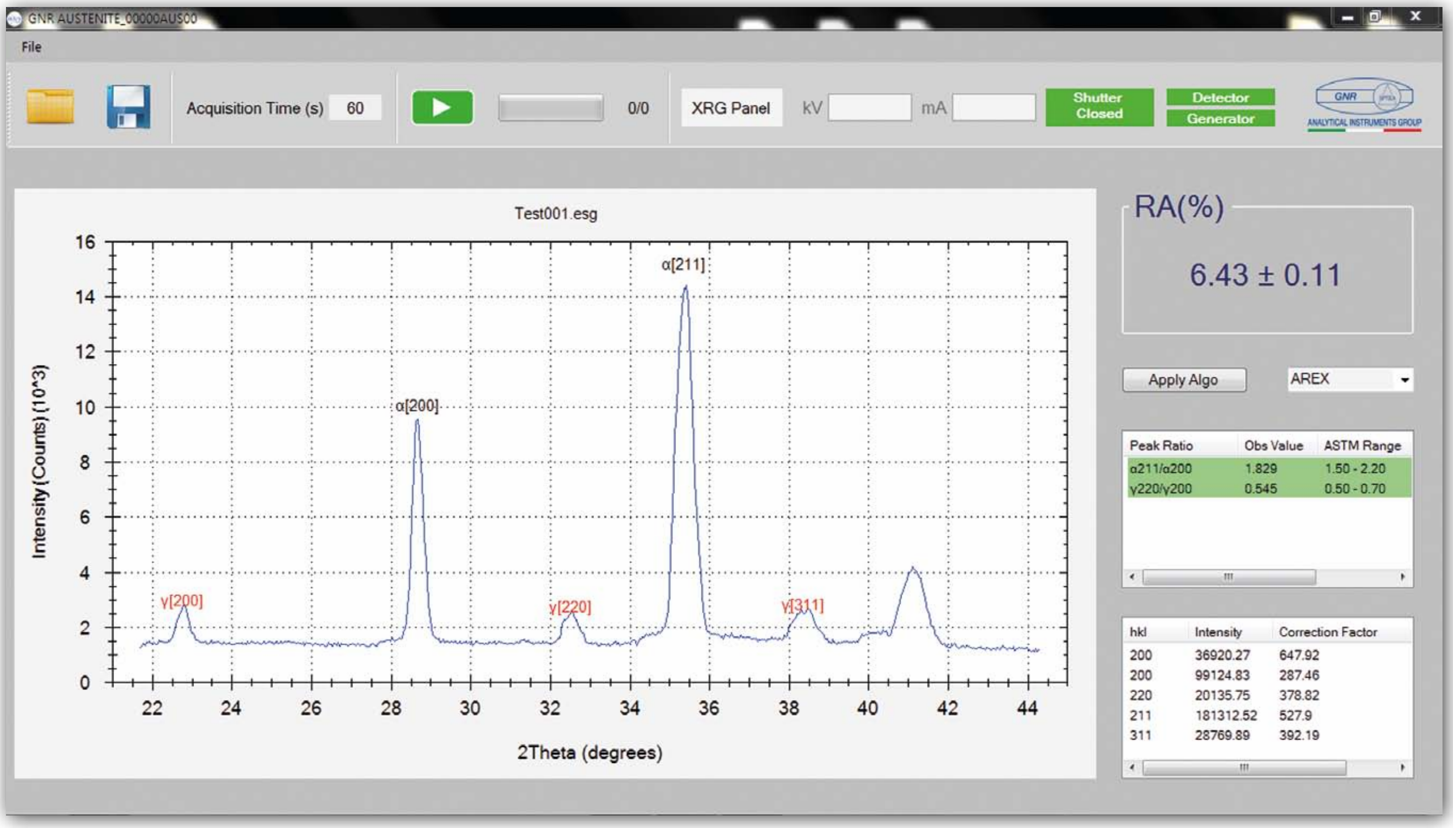


Retained austenite measurements on specimens in different points, representative of the overall thermal treatment cycle involving Carburizing, Hardening, Tempering and Blasting (CHTB)

Software

Hardware control, data acquisition and analysis are managed by AUSTENITE software in a user-friendly interface. At the end of each measurement, the volume % value of retained austenite is immediately displayed as well 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”).

Custom data analysis are available on request.



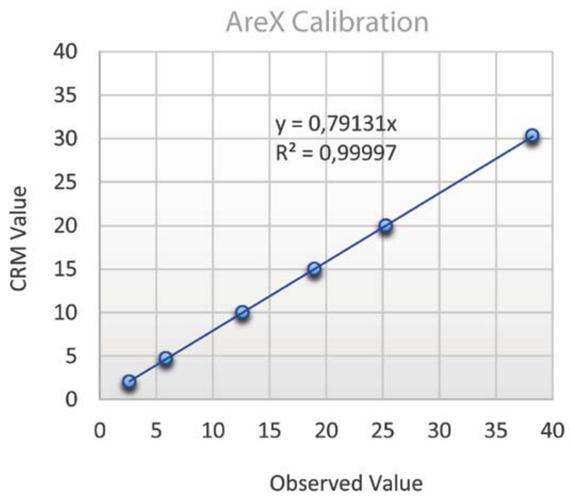
Austenite Software

ArexX Product Specifications

The accuracy of the retained austenite determination is challenged at very low austenite contents (~1 weight %).
XRD has been used to measure the amount of Retained Austenite in steel. The standard method (ASTM E-975-03) employs integrated intensities ratios of austenite and martensite or ferrite phases.
Usually Retained Austenite determination can be better performed by XRD than optical microscopy (OM) because it is user-independent. Using OM method, underestimation is a frequent issue.

GNR ArexX is a Θ-Θ diffractometer dedicated to retained austenite determination. Its high-count statistic involves an error on the single measurement in the 0.02-0.03% range.

GNR ArexX precision is in the 0.10-0.60% range on three measurements.



GNR ArexX accuracy is in the 0.05-0.50% range 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

Sample Preparation

GNR ArexX allows analyzing both specimens and real samples.

Samples for X-ray diffraction austenite analysis must be handled without heating them; if adequate cooling is not used, heat effects can transform retained austenite into other phases. Saw cutting rather than abrasive wheel cutting is recommended for sample removal.

Standard metallographic wet-grinding and polishing methods shall be used to prepare sample for X-Ray diffraction analysis.

Sample size must be large enough to contain the X-ray beam

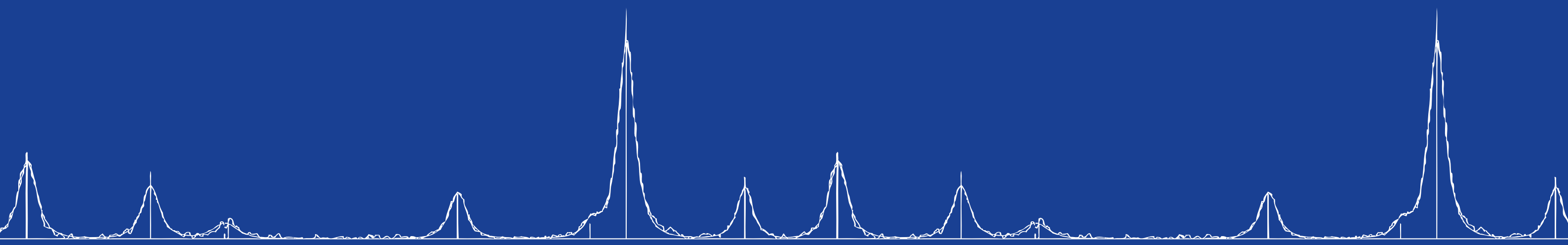
Useful advice on sample preparation can be found in E 975-03 and SAE 453.

X-Ray Generator	Output stability	< 0.01 % (for 10% power supply fluctuation)
	Max. output voltage	60 kV
	Max. output current	60 mA (option: 80 mA)
	Voltage step width	0.1 kV
	Current step width	0.1 mA
	Ripple	0.03% rms < 1kHz, 0.75% rms > 1kHz
	Preheat and ramp	Automatic preheat and ramp control circuit
	Input voltage	230 Vac +/- 10%, 50 or 60 Hz, single phase
X-Ray Tube	Type	Glass Mo anode (option: Ceramic Mo anode)
	Focus	0.4 x 8 mm FF
	Collimation	Monocapillary collimator: 1 mm diameter
	Max. output	3.0 kW
Configuration	Scanning angular range	22° < 2Theta < 44°
Sample Holder	Dimensions	110 mm x 150 mm
Case	Dimensions	658 W x 1059 H x 762 D (mm)
	X-rays leakage	< 1 mSv/Year (full safety shielding according to the international guidelines)
Processing Unit	Computer type	Personal Computer, latest version
	Items controlled	X-ray generator, detector, counting chain
	Basic data processing	Creation of calibration curves, Retained Austenite quantification

Safety Assurance

AreX complies with the statutory requirements regarding X-Ray, machine and electrical safety.
Maximum X-Ray safety with radiation level significantly below the annual dose limit for general public (1 mSv/year) following ANSI N43.3 - 1993 and other industry standards for open beam X-Ray operation.

The radiation enclosure door cannot be opened when X-Rays are on and the system immediately switch off if shutter is forced to open. This function completely protects user from radiation exposure.



Local Agent

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