GNR OPTICA

ANALYTICAL INSTRUMENTS GROUP

AreX L

Retained Austenite Analyzer

www.gnr.it

About us

GNR, thanks to its 40 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.

GNR 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.

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

YouTube

GNR designs and produces X-Ray Diffractometers (XRD) and X-Ray Fluorescence Spectrometers (XRF) for the study of material structure and elemental composition for both academic and industrial applications.

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.

GNR periodically organizes at its facility courses and training for technicians and agents as well as seminars and demonstrations.

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


CERTIFICATO

Nr. 50 100 4258 Rev.006

IL SISTEMA DI GESTIONE PER LA QUALITÀ DI
THE QUALITY MANAGEMENT SYSTEM OF

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

SEDE LEGALE E OPERATIVA:
REGISTERED OFFICE AND OPERATIONAL SITE:

VIA TORINO 7
IT - 28010 AGRATE CONTURBIA (NO)

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

UNI EN ISO 9001:2015

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

Progettazione, fabbricazione, commercializzazione, 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

Design, manufacture, trade, service and start-up of optical emission spectometers and x-ray instruments for materials characterization. Trade of complementary and similar product

ACCREDIA

SGQ N° 049A

Francesco Scarlata

PRIMA CERTIFICAZIONE / FIRST CERTIFICATION: 2004-05-05

TÜV Italia • Gruppo TÜV SÜD • Via Carducci 125, Pal. 23 • 20099 Sesto San Giovanni (MI) • Italia • www.tuvsud.com/it

ZERTIFIKAT ◆ CERTIFICATE ◆ СЕРТИФИКАТ ◆ CERTIFICADO ◆ CERTIFICAT

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 ones will always be those shown in the final order confirmation.

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 975-13 Standard Practice for X-ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation” along with the AreX L 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 L**, 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.

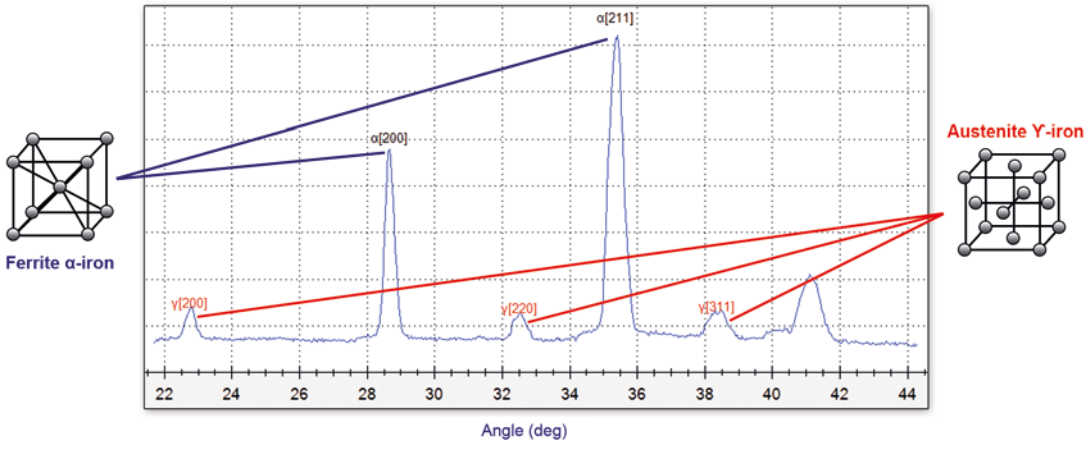
The integrated intensities of the austenite and the ferrite diffraction peaks are measured on the **GNR AreX L** diffractometer, providing four austenite/ferrite peak intensity ratios.

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 [%]** $\propto \frac{I_\gamma}{I_\gamma + I_\alpha}$



AreX L

Retained Austenite Analyzer

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 975-13**.

GNR AreX L 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.

Features

- Mo Anode X-Ray source with monocapillary collimation
- Power 1000 W
- Zr filter
- Angular range 21.5° - 44.5° (2-Theta)
- Z stage with laser alignment
- DECTRIS MYTHEN2 R 1D microstrip detector
- USB video camera for easy and precise sample alignment
- Sample holder dimensions 200 W x 130 D x 100 H mm
- Austenite software in a user-friendly interface
- Door interlock, thick steel frame and lead shield for x-ray safety protection
- Dimension: 660 W x 800 D x 1080 H mm
- Typical measuring time 60 seconds
- Custom solutions available

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

YouTube

Main unit

GNR AreX L Main Unit contains the following components:

- Power Supply
- HV Generator
- Electronic control unit

X-Ray source

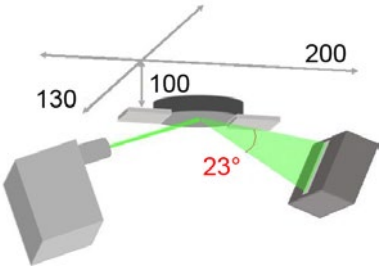
GNR AreX L Unit is equipped with a Mo X-Ray Tube. According to the test carried out with different anodes, the Mo source has been established as the optimal one because it allows to detect a large number of diffraction peaks, minimizing texture effects and because it has the best intensity response in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

Z-stage

GNR AreX L is equipped with a 0-10 mm manually variable Z stage with laser alignment and digital display control.

Sample holder

GNR AreX L sample holder allows to measure flat, concave and convex samples thanks to a laser control and a Z-stage alignment.



Linear position sensitive detector

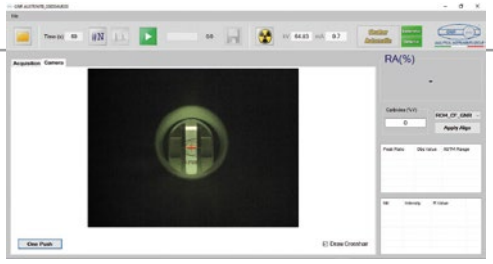
GNR AreX L is equipped with a Multi Strip Detector. GNR adopts DECTRIS MYTHEN X-Ray Detector.

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.

USB video camera

A professional USB video camera is mounted inside of **GNR AreX L** system and displays the measurement area on the sample surface.



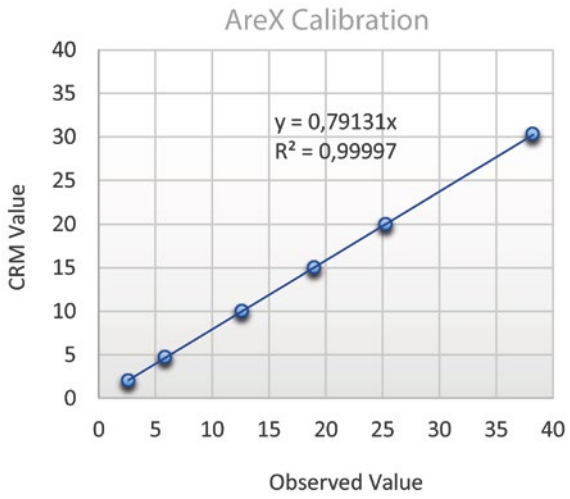
AreX L Product Specifications

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 975-13) 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 AreX L 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 AreX L precision is in the 0.10-0.60% range on three measurements.

GNR AreX L 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 $_\gamma$ 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 AreX L 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 975-13 and SAE 453.

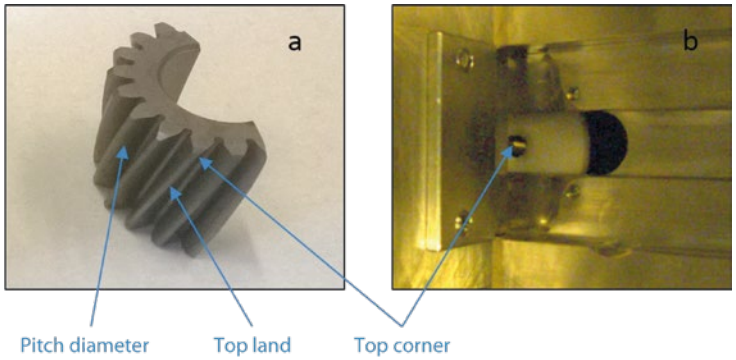
Retained Austenite in Gear Manufacturing

In this specific case, Retained Austenite amount has been determined by using the dedicated diffractometer **GNR AreX L** compliant with the standard practice **ASTM E 975-13**.

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

AreX L Instrumental Set Up	
Source	Fine Focus
Voltage [kV]	Mo 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]	60-120



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

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.

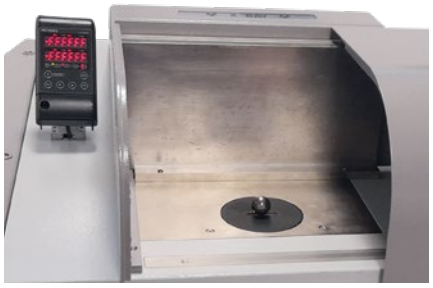
SAMPLE	Measuring Point	RA %				
		M1	M2	M3	Average	SD
Big Gear	Pitch Diameter	15.07	14.21	14.28	14.52	0.39
	Top Land	23.21	23.75	23.28	23.41	0.24
	Top Corner	26.04	25.81	26.27	26.04	0.19
Small Gear	Pitch Diameter	13.45	14.49	13.85	13.93	0.43
	Top Land	20.13	20.84	19.94	20.30	0.39
	Top Corner	24.69	25.15	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 L** precision is in the 0.10 - 0.60 range on three measurements.

AreX L for retained austenite measurement of rolling bearing spheres and cylinders

Bearings consist of rolling elements (balls, cylinders or barrel shapes) and rings, which form the raceways.
Bearing steels are heat treated to get martensitic microstructures providing high hardness necessary for good rolling contact fatigue performance. Without specific action taken, austenite is generally retained after heat treatment in the final component with a more or less important content.
Depending on the requirements of each application, retained austenite can be desired because of beneficial effects such as improvement of rolling contact fatigue performance, mostly in contaminated lubricating conditions, or can be avoid dimensional stability is needed for example in cases of bearings operating for long times at high temperatures.
Because bearing steels are subjected to more and more demanding operating conditions, heat treatments and microstructures are engineered in terms of retained austenite in order to use the beneficial effect of a stabilized retained austenite, or on the contrary to suppress retained austenite.
It is hence important to have a fast accurate and reliable tool to measure the Retained Austenite.
Metallographic methods by chemical etching and microscope evaluation can be very erratic, where traditional XRD measurements can be very time consuming and with severe constrains regarding sample positioning.
AreX L is overcoming all these constrains makes XRD measurements easier than ever.
To measure RA on spheres and cylinders it is necessary to overcome the geometrical constrains and ensure at the same time the most precise positioning of the specimens.
When it is intended to measure routinely specimens of different diameters AreX L has been proved to be the most suitable solution available today on the market.
A properly sample positioning device coupled with specifically designed sample holders allows to measure specimens of any diameter.

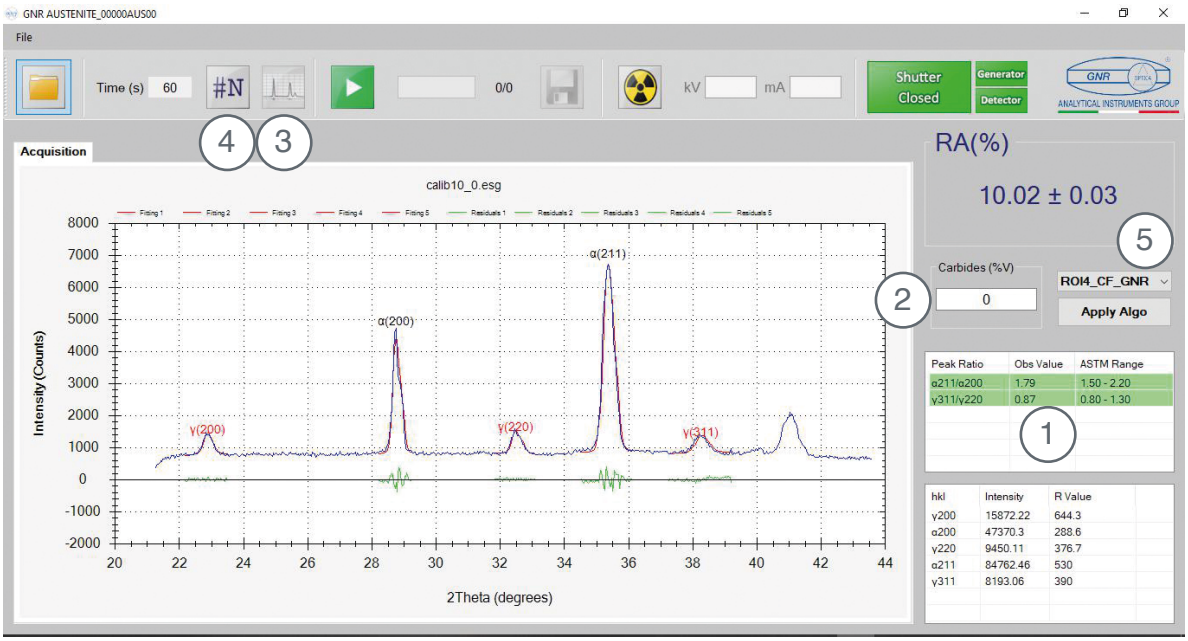


Once the sample is inserted into the sample holder, closing the door the laser automatically measure the specimen surface position that can be manually aligned by mean of a knob linked to a z stage.
The sample now is in position and the measurement can be performed.

The sample holders can be easily replaced to measure a specimen with different diameter repeating the alignment procedure. This process is done within seconds.
Specimen holders for cylinders as well as for other specimens are available or can be prepared as per customer's request.

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 975-13 (“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

1. Near random crystallographic orientations check
2. Carbide amount input
3. Manual peaks
4. Multiple measurements
5. Algorithms

The basic algorithm use multiple ROI (region of interest) approach (single peak analysis).
Advanced algorithms use full profile approach (simultaneous peak analysis: fitting of all collected data at once).

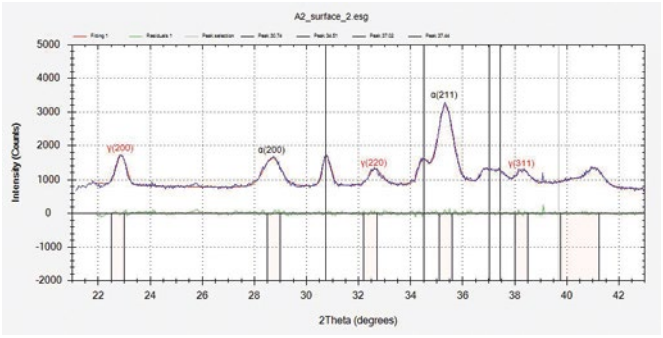
It can happen that carbide peaks are interfering with ferrite or austenite peaks. In this case, the austenite calculation can be less accurate.
To overcome this problem, the carbides feature has been introduced in the software. This will allow to select manually the carbide peaks and exclude their area in the calculations. This feature must be activated each time the diffractogram shows the presence of carbide peaks interfering with ferrite or austenite peaks.

For samples presenting preferred orientation is recommended to use the algorithm that performs a full profile fitting FP of the diffractogram, in this way we will obtain a value of retained austenite which takes into account the presence of texture in the material. Note that for the application of this algorithm it is necessary to collect three reflections for each phase. Note also that this algorithm can be used also for diffractograms of non-textured materials: in this case, the RA % value will be very similar to the one obtained with the usual ROI algorithm.

Wearing

Overlapping peaks from interfering phases as carbides: adding peaks manually

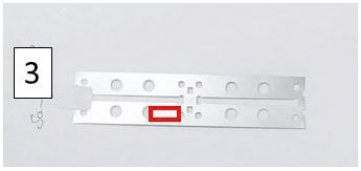
Sample	RA% (ROI)		RA % (Carbides correction)		delta
	average	st dev	average	st dev	
1	11.27	0.44	12.30	0.30	-1.30
2	9.09	0.42	11.89	0.32	-2.80
3	9.75	0.45	12.58	0.31	-2.83

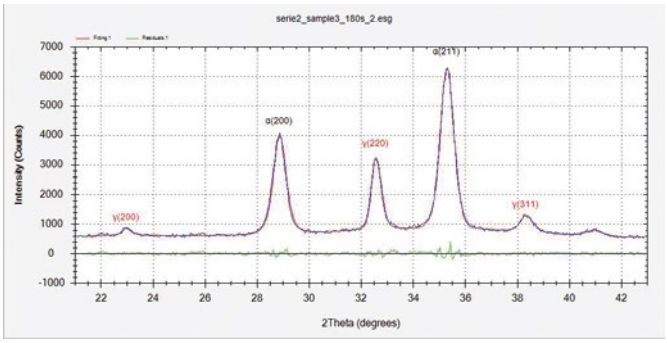


Razor Blades

Non-random oriented samples: advantage of collecting multiple peaks

Sample	RA% (ROI)		RA % (texture correction)		delta
	average	st dev	average	st dev	
1	15.79	0.34	10.14	0.76	5.65
2	20.98	0.55	14.66	0.32	6.32
3	12.11	0.35	8.44	0.85	3.66
4	15.20	0.10	10.92	0.57	4.28
5	11.21	0.08	7.39	0.42	3.82
6	21.60	0.30	15.00	0.25	6.60





X-Ray Generator	Output stability	< 0.01 % (for 10% power supply fluctuation)
	Max. output voltage	60 kV
	Max. output current	60 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
	Focus	0.4 x 8 mm FF
	Collimation	Monocapillary collimator: 1 mm diameter
	Max. output	3.0 kW
Configuration	Scanning angular range	21.5° - 44.5° (2-Theta)
Z-stage	Range	0-10 mm with laser alignment
Sample Holder	Dimensions	200 W x 130 D x 100 H mm
Case	Dimensions	660 W x 800 D x 1080 H 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 L 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.

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