AreX Diffractometer

GNR Proposal for measuring Retained Austenite in the industrial domain and in laboratory

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What is Retained Austenite?

Austenite is a phase of iron stable at high temperature. Hardening of steels requires heating to an austenitic phase and quenching to room temperature to ferrite and martensite.

Austenite that does not transform to martensite or ferrite upon quenching is defined retained austenite.

Depending of the steel chemistry and specific heat treatment, the retained austenite level in the case can vary from over 50% of the structure to nearly zero.

How does Retained Austenite affect the properties of a component?

Austenite is a very useful structural constituent of advanced high-strength steels.

Its ability to strengthen in many different ways offer possibilities to obtain a unique range of mechanical and technological properties.

High Percentage of Retained Austenite can reduce:

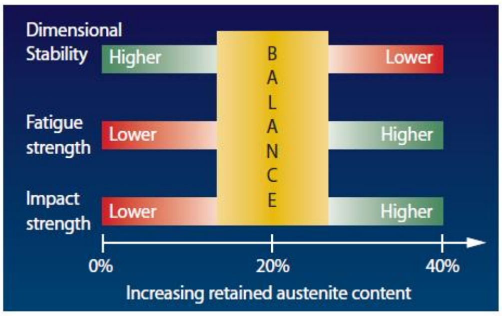
- Elastic limits
- Hardness
- High Cycle Fatigue Life
- Dimensional stability
- On the contrary low % of Retained Austenite can reduce:
- Fracture Toughness
- Rolling Contact Fatigue Life
- Low Cycle Fatigue Life

It is important to recognize that a balance must be created between the mechanical properties of a component and the optimum percentage of retained austenite for a given application.

Retained austenite is highly undesirable in components for the tool industry; Retained Austenite is recognized as a major cause for premature failure.

The low hardness of Retained Austenite is incompatible with the most applications that require the maximum attainable hardness to resist wear. The bearing and gear industries have a more favourable view toward having some percentage of retained austenite (from 5 to 30%), <u>in some cases even lower than 5%, limit that can be achieved by AreX, thanks to its LOD of 1%.</u>

Sample: Retained Austenite affects the properties of bearing steels



By controlling the level of Retained Austenite, its beneficial effects can be realized without suffering from its negative influences. To obtain the optimum level of Retained Austenite requires a delicate balance of controls and mast take into account such items as material chemistry and heat treatment process variables.

Controlling the Retained Austenite amount at different steps of manufacturing process and on the final product is very important to ensure product quality and to optimize cost of production process.

How Retained Austenite is measured?

In several laboratories and production plants, Retained Austenite is still measured by metallography using an Optical Microscope.

Unfortunately, there are several constraints in the application of this analytical method to the industrial segment.

First-of-all, it is a destructive technique and it can be used to determine austenite content only if a sufficient quantity is present. In many steels, 10 to 15 percent of retained austenite is the minimum detectable limit.

Then, the Optical Microscopy (OM) analytical method is user-independent and underestimation is a frequent issue.

Moreover, it could be very difficult to measure Retained Austenite in austenite martensite mixed microstructures.

Another potential method to analyse Retained Austenite is the adoption of the X-Ray Diffraction.

According to the new research, X-Ray Diffraction is considered the most accurate method of determining the percentage of Retained Austenite in Steels. X-Ray Diffraction technique is commonly non-destructive and can precisely measure the retained austenite concentration as low as 0.5%.

ASTM international to address this specific method has released a standard practice (ASTM E 975-03) and this is the reference in the industrial sector for any kind of quality control.

Obviously, a traditional powder or a multi-purpose diffractometer could be adopted for the analysis of Retained Austenite; unfortunately, end user needs to have a sample as a specimen with small dimensions and the acquisition time and elaboration could be around one hour for single measurement.

Clearly, this could fit for a requirement in a laboratory with few pieces to be analysed.

Not properly if the end user needs to analyse for quality control all the components of a specific batch in the manufacturing process.

Moreover, the level of usage of standard diffractometer in many cases is not compatible in the process line, in terms of geometrical typology of samples to be measured, in terms of rapidity of the analysis and in terms of knowledge of operator.

This is the reason why GNR develop AreX, a dedicated Diffractometer for Retained Austenite measurement, specifically manufactured for the industrial requirements.

Why AreX is the most suitable solution to measure retained austenite?

AreX system is a fixed-angular range X-Ray diffractometer equipped with the most advanced technical features, which grant accuracy, precision, safety and easiness of use, specifically designed for quantitative determination of Retained Austenite.

Accuracy and Precision

- AreX is compliant with ASTM E 975-03 standard practice
- AreX has an accuracy better than 0.5%, guaranteed by CRM calibration
- AreX has a standard deviation over ten repetition better than 0.6%.
- AreX has a limit of detection of 0.5% of RA.

Rapidity

- AreX is a simultaneous diffractometer and the analysis is performed in less than three minutes, depending on samples dimensions and geometry. Typical measuring time is 60 seconds
- AreX is very easy and fast to analyse the sample on several points thanks to the positioning by USB video camera

Easiness of Use

- Operator does not need to have specific x-ray diffraction knowledge and competence; just put the sample on the equipment and launch the measurement … AreX allows you to obtain the result with a click of a mouse
- AreX is the most suitable solution for quality assurance and quality control also in production line

Sample Typology

- AreX allows measuring both specimens and real samples
- If the End User asks for analysis of components of specific dimensions, a custom solutions can be provided by GNR

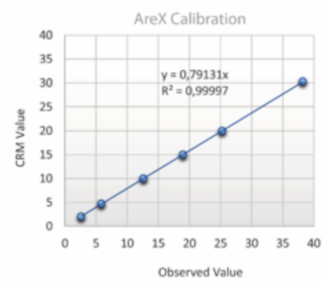
<u>AreX Features</u>

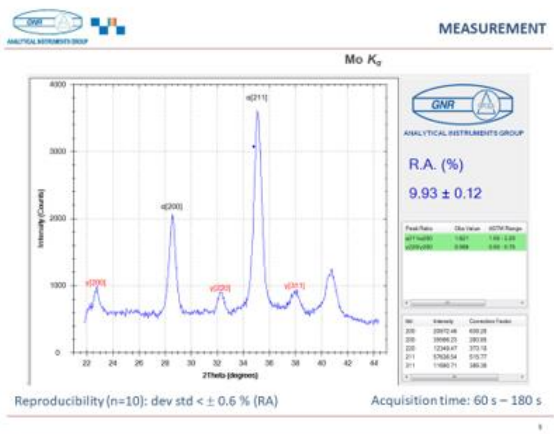
- Compliant with ASTM E 975-03
- Fast Measurement (< 3 minutes)
- 3000 W X-Ray Generator
- Mo X-Ray Tube with Monocapillary Collimation
- Zirconium Kβ filter
- Fast Detector
- Sample Holder (110 x 150 mm)
- Custom Solutions Available
- External Cooling System

AreX (658 x 762 x 1059) mm; 100 kg

AreX: Custom solution for big samples

Num	RA %	
1	5.2	
2	5.93	
3	4.88	
4	5.45	
5	5.34	
6	5.63	
7	4.86	
8	4.73	
9	4.56	
10	5.35	
	5.193	Average
	0.43	Std Dev





To calculate the volumetric percentage of retained austenite up to seven diffraction peaks can be collected by AreX, three for ferrite/martensite phase and four for austenite phase.

A comparison of the intensities of the four peak yields the volume percent concentration of Retained Austenite in the sample.

Which is the sample preparation needed to use AreX?

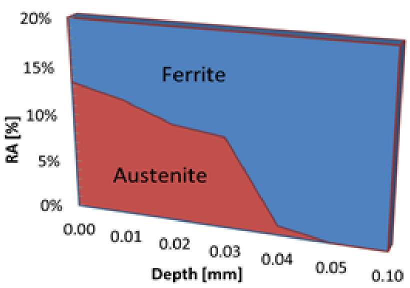
- Real Sample can be analyses “AS IT IS” in the most cases
- Standard metallographic wet-grinding and polishing methods can be used
- For surface lapping 6 to 0.2 µm by diamond or alumina paste is the right choice
- Saw catting and electrolytic polishing machine can be used
- Is absolutely better to avoid abrasive wheel cutting and hot acid etching

- Standard Practice ASTM E 975-03 describes the sample preparation procedure to be followed

Volume of Analysis and Depth Profile Analysis

The value of Retained Austenite is measured in a volume of an ellipse 4 mm x 2 mm and 30-micron depth.

It is also possible to perform a depth profile analysis after cooled mechanical removal.



Depth (mm)	RA [%]
0.00	13.45 ± 0.35
0.01	12.19 ± 0.42
0.02	10.20 ± 0.32
0.03	9.51 ± 0.06
0.05/0.10	< 1

AreX: an innovative solution

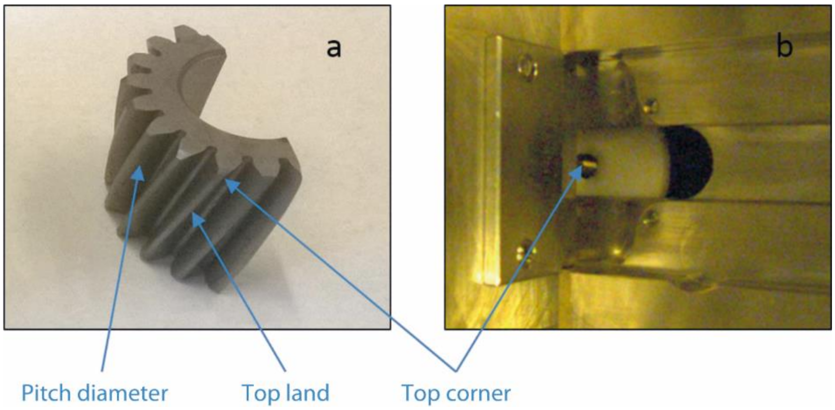
AreX is an innovative solution for the following production player

- Bearing and Gear Industries
- Heat Treatments Companies
- Automotive industries (gear boxes, transmission)
- Material and Failure analysis Laboratories

In general, in all the industrial companies or laboratories where:

- Retained Austenite is measured
- Quality Control of the components is crucial (by internal procedure or for external compliance)
- High Volume of Analysis
- Failure and Mechanical Analysis is performed

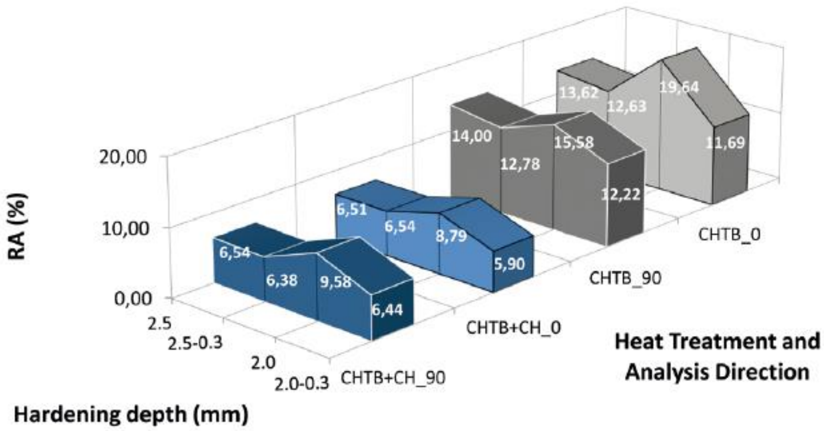
Real Case: Retained Austenite in Gear Manufacturing



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

Each measurement has been repeated three times (M1-M3).

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



	Optical Microscope	X-Ray Diffraction	
		Standard Diffractometer	AreX Solution
Volume	▪ Few Specimens per Day	▪ Max 10/15 Specimens per Day	▪ 100 Specimens or Real Samples per Day
Sample Typology	▪ Specimens after Metallographic procedure	▪ Specimens and Small Real Samples	▪ Both Specimens and Real Samples
Quality	▪ User-Dependent ▪ High LOD (10 – 15%)	▪ User-Independent ▪ LOD 0.5%	▪ User-Independent ▪ LOD 0.5%, SD < 0.6%, Acc. < 0.5%
Rapidity	▪ Dependent on user expertise to prepare the specimens and read the image in the Optical Microscope	▪ From 30 to 60 minutes each measurement (as average)	▪ 3 minutes max ▪ Typically 60 seconds each measurement
Easiness of Use	▪ Dependent on user expertise ▪ Expert User in Optical Microscope is required ▪ Typical Activity performed in Laboratory	▪ Sample Positioning and Software Elaboration ▪ Expert User in Diffraction is required ▪ Typical Activity performed in Laboratory	▪ Very easy: just a click of a mouse ▪ Standard Operator can use the Instrument, also in the production line ▪ No Diffraction expertise is required