

Retained Austenite Benefits or Avoidance Requires Dependable Determination

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Abstract

Retained austenite can be disastrous or increase the life of heat-treated components. However, discussions on the right amount of retained austenite are only meaningful when measured correctly. The proper determination in microstructures samples requires a trained metallurgist and remains to be very subjective to the individual's eye. Company standards of microstructure tables are used as individual solution. However, factual results are only achievable with the use of an x-ray diffractometer. This paper discusses the practical experience of metallurgical results and shows state of the art solutions as today's standard.

Introduction

Retained austenite is that fraction of austenite which remains untransformed at the end of the hardening process. Retained austenite is considered detrimental or undesirable in most cases but there are certain applications where some amount of retained austenite is considered as desirable. Let us consider both of these cases.

Retained austenite strongly influences the properties of steel. The fatigue life, ductility, toughness, hardness, yield strength, and machinability all depend on austenite content.

Accurate measurement of the volume percent retained austenite is of critical importance to the optimization of heat treatment procedures. Austenite, being an unstable phase at room temperature, will transform to martensite during use, causing brittleness and an increase in volume potentially leading to failure of critical components.

However, the accurate measurement in manufactured steels remains a challenge as commonly used visual metallurgical sample investigations are subjective and mostly provide a very false reading, magnetic measurements need part specific calibration, Electron Back Scattering (EBSD) measurements require expensive equipment, intensive sample preparations and long measurement times.

New developments in X-Ray equipment provide measurements in minutes and can also compensate for the influences of carbides in high carbon steels or texture orientations in rolled sheet metals.

Basics of Retained Austenite

Austenite, also known as gamma-phase iron (γ -Fe), is a metallic, non-magnetic allotropic form of iron and can be described also as a solid solution of Iron with some alloying elements.

In plain steels as example, austenite exists above the critical eutectoid temperature 727°C or thousands of other steel alloys have different eutectoid temperatures. Austenite can exist at room temperature in stainless steels or in any other steel as not transformed austenite otherwise called retained Austenite.

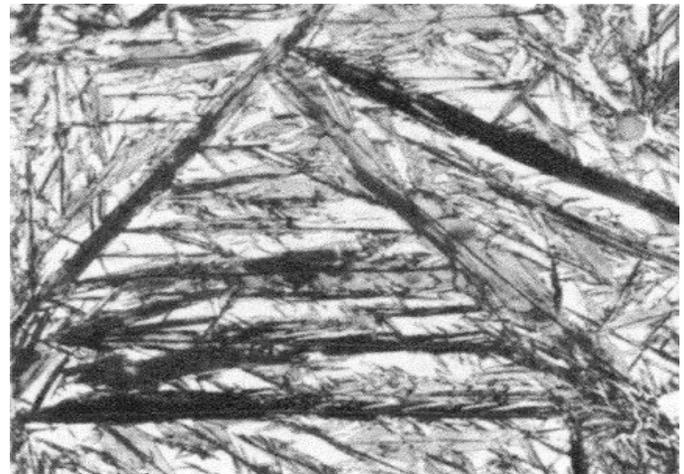


Figure 1: Retained Austenite (white) with plate Martensite in a carbon steel, at 1000 times magnification, etched with Nital solution. (1)

Retained Austenite Creation

Hardening of steels requires heating to an austenitic phase and quenching to room temperature to produce a hard martensitic phase. Austenite is an FCC phase that is stable above a temperature of 727 C. Due to incomplete transformation during quenching some austenite is retained at room temperature. Retained austenite can dramatically decrease the mechanical properties of the steel. Properties such as fatigue strength, toughness, hardness, yield strength and machinability can be influenced by retained austenite.

Austenite can transform in service as a result of thermal cycles, plastic deformation, or shock. Shot peening, for example, will transform the austenite on the surface of gear teeth. Exposure to extreme cold renders the austenite increasingly unstable as the temperature diminishes. The transformation of austenite to ferrite involves a nominal 4% volume increase. A linear dimensional increase on the order of the cube root of that would lead to seizure and excessive interference in precision gearing and bearings. Accurate measurement of the retained austenite levels is important in the development and control of a heat treatment process.

Austenite transforms to Martensite between the M_s and M_f temperatures. However, this transformation never goes to completion for carbon contents higher than 0.25 wt pct, i.e. 100% Martensite. Figure 2.

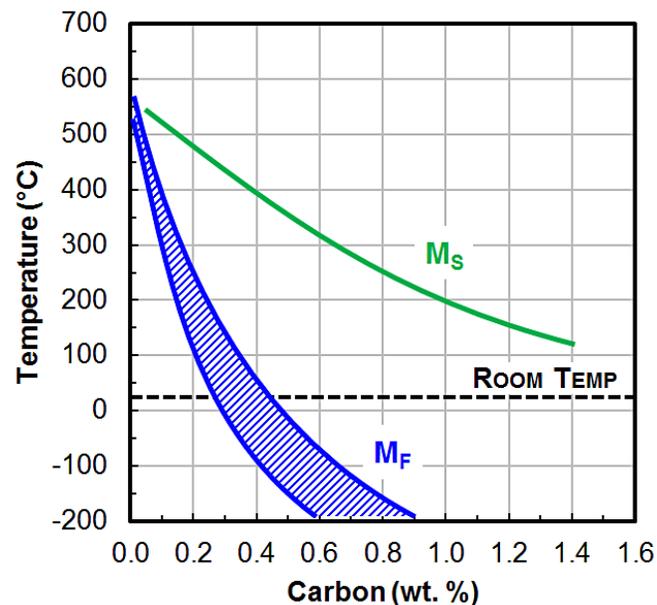


Figure 2: Martensite start (M_s) and finish (M_f) temperatures as a function of carbon content for plain carbon steels. (2)

The M_s and M_f temperatures are lowered by most alloying elements and an increasing austenitizing temperature but mostly by increasing the carbon content as seen in Figure 3.

Higher austenitizing temperature brings more carbon and alloying elements into solution in austenite. Also, this increased temperature results in more thermal stresses on quenching, which oppose martensitic transformation, i.e. both factors increase retained austenite.

The deformation of austenite above a temperature called M_d (higher than M_s), lowers M_s temperature resulting in increase of retained austenite. This untransformed austenite is called retained austenite. For example, steels with carbon less than 0.25%, when quenched to room temperature have little retained austenite, because room temperature is below the M_f at such carbon contents in steels.

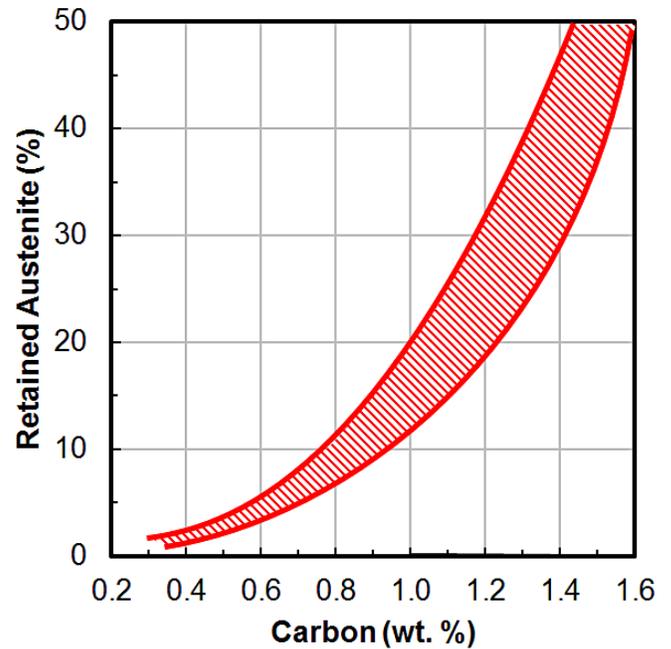


Figure 3: Retained austenite as a function of carbon content for plain carbon steels quenched to room temperature. (2)

The amount of retained austenite increases with the increase of carbon content of the steel.

Why is the measuring of Retained Austenite of interest?

Retained Austenite is the result of an unfinished Martensite transformation after quenching of high carbon steel after austenitizing. Resulting in:

Lower hardness: Austenite being a softer phase, if RA% is high, you will observe soft spots. The soft spots are detrimental in applications such as cutting tools where wear resistance is one of the most important factors affecting the service performance and life.

Volume change: The transformation of austenite to martensite is accompanied by increase in volume. Therefore, retained austenite, when it transforms to martensite during the service life of the component, would affect the dimensional stability of the component. This is detrimental in applications such as bearings and gauges where dimensional stability is extremely important.

Premature Failure: The fraction of austenite is retained (untransformed) at the end of the hardening process is likely to transform to martensite during subsequent tempering or when the hardened part undergoes stress and strain in service. This newly transformed martensite will remain un-tempered and hence cause brittleness, which can cause premature failure of the component in service. Due to this reason, retained austenite is detrimental in applications such as tools and dies, where high impact loading is an essential service condition.

Increase fatigue resistance: Finely dispersed retained austenite resists the propagation of fatigue cracks and improves rolling contact fatigue (RCF) stress. Therefore, some amount of retained austenite is considered to be beneficial in certain applications. One such application is bearings working with contaminated lubricants (eg: railway bearings). In some cases, bearing components made of through hardening steels like SAE 52100 are carbonitrided because the carbonitriding process gives higher surface hardness and increases wear resistance and it also promotes retained austenite. (3)

Measuring Retained Austenite

The volume fractions of phases in materials have usually been evaluated by optical microscopy, magnetic analysis, and X-ray diffractometry. Among these methods, the X-ray diffractometry method is one of the most efficient means (4).

The accuracy of the techniques used to identify and measure austenite in steel decreases significantly with decreasing amounts of austenite. As RA fractions become small, the morphology makes measurement difficult (e.g., thin RA films between martensite plates or laths (5)). The most common measurement techniques are:

- Visually with a Microscope
- Magnetic, whereas Martensite is ferro Magnetic and Austenite is Paramagnetic
- EBSD (Electron Backscatter Diffraction)
- X-ray diffractometry (XRD) measuring different lattice structure of the Martensite (BCT) and the Retained Austenite (FCC)

Microscope

- Destructive
- Typical measure area $>100\mu\text{m}^2$
- Subjective measurement
- Variance up to 20%, depending on the RA content and tempering level

Measuring Retained Austenite with a light microscope is performed on a metallographic prepared (cut, ground, high polished) and etched microsample at a e.g. 500 times magnification.

The proper segmentation of the sample out of the part e.g. gear is very crucial to receive a representative quantity of the measured area. The mechanical force and heat introduction during segmentation must be minimized to avoid the transformation of austenite. The removal of surface layer during grinding and polishing of the micro sample may also change the evaluation plane. The right etchant must be selected for the material (3-5% Nitric acid in alcohol is commonly used) and the etching time and intensity can vary the contrast of the

structure, in a way, that un-tempered (tetragonal) martensite plates cannot be differentiated from the residual unetched retained austenite.

Figure 4 shows plate Martensite next to Retained Austenite in a 1.31% C, 0.18% Si, 0.2% Mn steel after 950°C austenitizing and water quenching. In the as quenched condition, the Martensite is light and the Retained Austenite darker as shown in the left picture (a).

After additional tempering at 150°C, the Martensite appears darker than the Retained Austenite due to the carbide precipitation. (6)

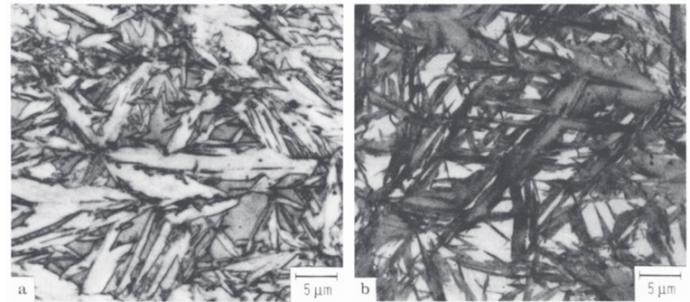


Figure 4: light un-tempered (left) and tempered (right) plate Martensite next to Retained Austenite. (6)

Magnetic

- Non-destructive
- Fast, reliable
- Median over the volume
- Typical measure volume $>1\text{ cm}^3$
- Measuring range 1...30%
- Variance 0.5 %, depending on the RA content
- Calibration necessary

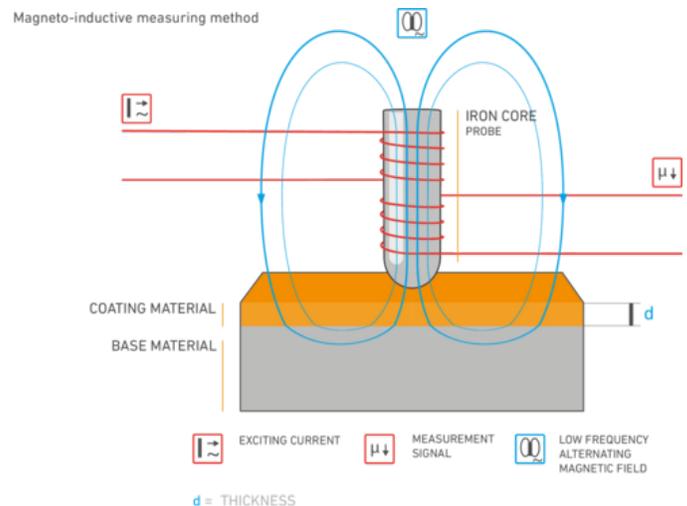


Figure 5: schematic of the magneto-inductive measuring method (7)

Measuring retained austenite with magnetic sensor

As illustrated in Figure 5, the sample will be magnetized to saturation and the saturation polarization will be measured. The difference between measured and theoretical saturation the retained austenite content can be calculated: $V_{RA} = 100Vol.\% - V_M \%$

Electron Backscatter Diffraction (EBSD)

- Destructive
- Very small measure volume
- Penetration depth 5...10 nm
- Variance 0.5 % RA content
- The sample will be (monochromatic) scanned in a SEM (Scanning Electron Microscope) and moved.
 - The bragged lines indicate grain orientation and local phase content.

An example of retained austenite detected by EBSD is shown in Figure 6.

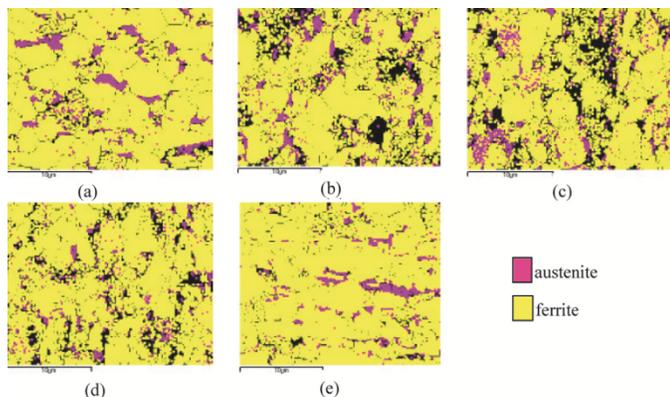


Figure 6: EBSD maps showing the effects of strain on retained austenite (a) 0%/6.3% RA (b) 5%/6.1%RA (c) 10%/5.4%RA (d) 20%/3.9% RA (7)

X-ray diffraction (XRD)

- Non-destructive
- Penetration depth 1...10 μm
- Variance 0.1 % RA content

X-ray diffraction, illustrated in Figure 7, is considered to be the most accurate method of determining the amount of retained austenite in steels.

It is a non-destructive analytical technique used to identify and quantify phases in a material. Every crystalline phase produces a characteristic diffraction pattern (e.g., fingerprint) as illustrated for steel in Figure 8.

The volume fraction can be determined by the X-ray diffraction since X-ray diffraction intensities are directly proportional to the volume of the phase considered.

The most severe shortcoming with this method is the problem of calibration due to preferred orientation, since all materials always have a preferred orientation to some extent. The problem of quantitative phase analysis in textured material is one of obtaining the integrated intensity averaged over all orientations of the specimen with respect to the X-ray beam. Methods of averaging involve randomizing the intensities by mathematical or mechanical means. Several methods of averaging the intensities have been proposed for the quantitative phase analysis of textured materials. (4)

Any electromagnetic radiation interacts with the material through either absorption and energizing the material system and expelling neutrons from atoms; or diffusion, during which the radiation is diffused by the matter and the electromagnetic waves associated with it change direction of propagation. This change can be accompanied by energy exchanges between photons and matter.

The technique of X-ray diffraction is based on coherent elastic scattering: the macroscopic phenomenon of diffraction arises from the coherent sum of all the electromagnetic waves diffused by the atoms found along the same family of reticular planes. To manifest itself, it necessarily requires the presence of a reticular order, as found in crystals or in crystalline materials.

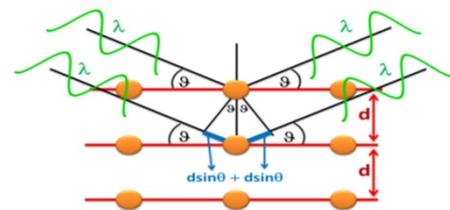


Figure 7: Scattering schematics

The incoming beam (upper left in Figure 9) scatters re-radiating a small portion of its intensity as spherical wave. If this happens symmetrically at a discrete distance d , the waves are in synchrony (constructive mode) only in the direction where their path-length difference $2d\sin\theta$ is equal to an integer multiple of the radiation wavelength λ . This creates a diffracted beam at an angle measuring 2θ producing a figure called diffraction pattern that can be collected and represented as follows in function of the detecting method.

In addition to phase analysis, X-ray diffraction can also be used to analyze microstructural features such as texture, residual stress and grain size. Texture produces systematic deviations of peak intensity from the characteristic diffraction pattern of a phase. The intensity deviation can be used to quantify the fraction of grains in a certain orientation by tilting and rotating the sample in the diffractometer as shown in Figure 9

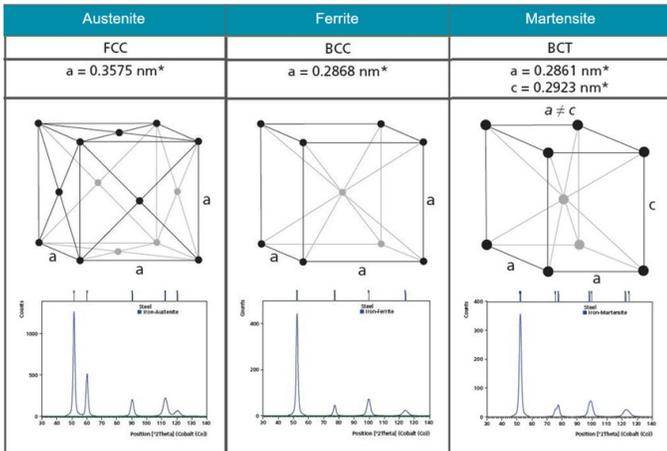


Figure 8: Crystal structures of Austenite, Ferrite and Martensite and their corresponding diffraction patterns

With the x-ray measurement, the diffraction plane is crucial: only the grain in the lattice n_{hkl} are registered.

Therefore the sample has to be turned and tilted and is measuring the intensity at multiple angle positions.

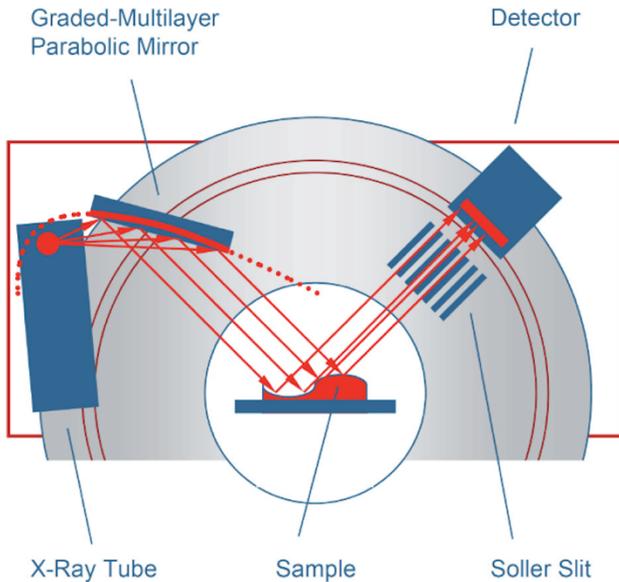


Figure 9: Schematics and rotating the sample while measuring with X-Ray

Carbide Correction

Carbide presence can have an influence on the determination of RA%Vol. The influence is given by the presence of overlapped or adjacent carbide peaks that can interfere and alter the intensity of the ferrite or austenite peaks. Standard algorithm based on the evaluation of the region of interest, can lead to an erratic evaluation of the RA%Vol since it can be totally or partially considered in the peak integration.

Using the Full profile approach, it is possible to consider the influence of this peak and do not consider it, as well as all the other peaks of carbides that can interfere.

Orientation

Measurements of retained austenite using x-ray diffraction are often employed despite the caveat that these methods only apply to uniform (random) texture distributions. Due to the strong crystallographic texture caused by deformation during processing, these assumptions are typically not valid for rolled sheet steel (e.g transformation induced plasticity (TRIP) steels) There is data to indicate that the transformation will not be distributed evenly as a function of orientation, but particular stress states will cause some orientations to transform at a higher rate than other orientations. A technique using complete pole figure averaging using neutron diffraction was developed to measure the retained austenite in textured TRIP steels as well as provide an estimate on the uncertainty in the phase fraction (8)

Comparing different measuring techniques

In the following we are comparing three very common applications and materials where precise retained austenite measurement is essential and most applied for the best performance: Carburized gears, bearings and transformation Induced Plasticity (TRIP) steels.

Carburized gears

Automotive gears are commonly carburized to .9% carbon content on the surface and oil quenched. Depending on the alloy and application, a retained austenite content of 25%+/-5% is often desired for high fatigue resistance. However, as the carbon content diminishes with the diffusion depth in the material, the retained austenite content is responding alike. See Figure 10

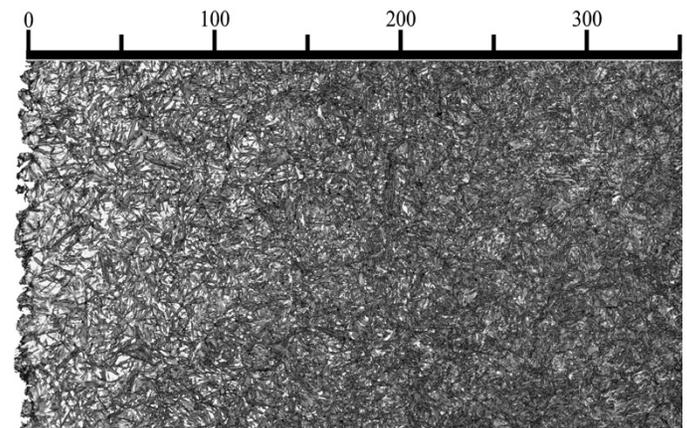


Figure 10: Diminishing retained austenite content in a carburized gear (depth in μm) (9)

Often a retained austenite content of 25% is desired in the root of a gear to achieve highest fatigue performance in automotive gears transmissions. Commonly a prepared and etched metallographic sample perpendicular to the carburizing zone is used to quantify the maximum RA content as shown in Figure 10. However, this method is subjective to the examiner and provides a large variation of results. Therefore, some companies developed own corporate standard micrographs. By removing the surface layer by layer with electro polishing and measuring the RA content with X-Ray and taking a microscope picture of the examined area. The results of such a reference guide can be seen in the appendix.

TRIP Steels

The overall phase fraction of austenite in TRIP steels is of interest as the rate of transformation is thought to strongly affect mechanical strength and ductility (8).

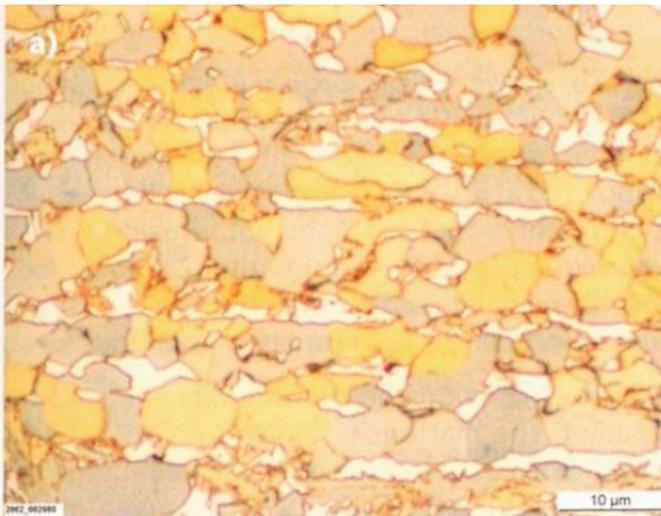


Figure 11: Ferrite (yellow), Bainite (brownish), Austenite/Martensite (white) (7) (11)

Table 1: Test results TRIP 700 material

No.	Method	Evaluation Method	Results RA %
1	XRD (Cr)	ASTM E975-3	10 - 12.9
2	XRD (Cu)	Rietveld	11.1
3	XRD (Cr)	Rietveld	13
4	XRD (Mo)	ASTM E975-3	16.2
5	XRD (Co)	Rietveld	18.9

6	XRD (Mo)	Rietveld	19.2
7	Magnet	Hoselitz Corr.	17.1
8	Magnet	Density Corr.	19.4
9	Magnet	Hoselitz Corr	22.2

Figure 11 is showing an example with Retained Austenite measured in a round robin test varying between 11 and 19% with X-Ray and 17 to 22% with Magnetic-Induction, as the RA is textured and not evenly distributed, (7). See Table 1

Bearings

With a carbon content of 1% and a chrome content of 1.5%, steels for bearings not all carbon is bonded in carbides, sufficient carbon remains in the matrix to form high amounts of retained austenite after quenching. Figure 12 is showing a typical AISI 52100 bearing steel microstructure after hardening at 840°C, followed by a deep freeze and tempering, showing a typical evenly carbide distribution within the fine dark etched martensite structure and small amounts, non-visible residual austenite. In a x-ray round robin test with various tube materials and evaluation methods, the results showed 11% RA with a much lower spread of a typical +/- 1.5% whereas Magnetic inductive measurements showed higher results of 14% +/-2% (Table 2)

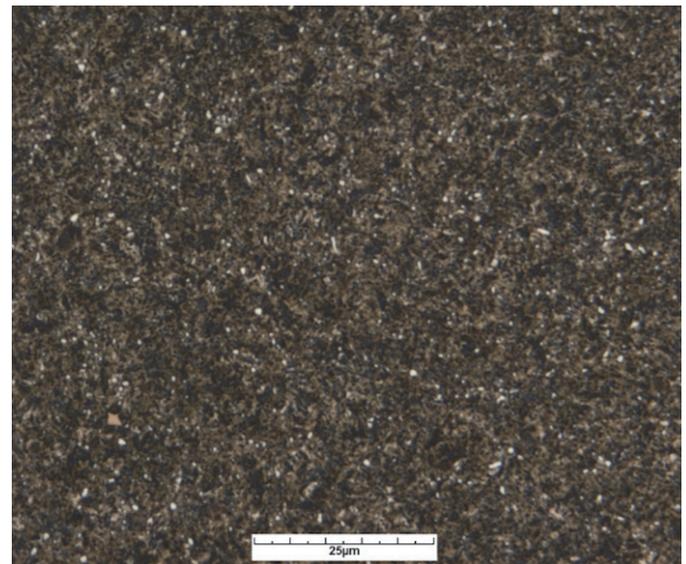


Figure 12: Tempered Martensite (dark), Carbide and Retained Austenite (White) (7)

Aside the great variations in some test results of various measurements techniques, the measured area and penetration depth (reference volume), the time to prepare the sample and the measurements itself, is playing a big role when applying RA measurement in the daily quality assurance routine. Table 3 is

showing a comparison of x-ray diffraction (XRD), Electron Back Scattering (EBSD), Magnet induction and Microscope measurements.

Table 2: Test results on AISI 52100 Material

No.	Method	Evaluation Method	Results RA %
1	XRD (Mo)	ASTM E975-3	8.5
2	XRD (Cr)	ASTM E975-3	10.4
3	XRD (Cr)	Rietveld	11.4
4	XRD (Cu-Ag)	Rietveld	12.3
5	XRD (Cr-Mo)	Rietveld	11.3
6	XRD (Co)	Rietveld	11.1
7	Magnet	Density Corr.	12.6
8	Magnet	5200 Corr.	16
9	Magnet	Hoselitz Corr	14.7

Table 3: Comparison of different measurement techniques

	XRD	EBSD	Microscope	Magnet
Prep time	10 min.	2 hr	1 hr	/
Measure time	5 min	6 hrs	30 min.	1 min.
Total Time	15 min	8 hr	1.5 hr	1 min.
Penetration	10 μm	5 nm	surface	10 μm

Conclusions

Retained austenite plays a significant role in the performance of heat-treated steel components. Data indicates even very small amounts of RA may be influential. The ideal RA depends on the alloy and its application (e.g. bearing, gears or TRIP steel). As a result, appropriate measurement techniques and process controls need to be implemented to deliver targeted performance.

Light Microscopy provides qualitative assertion with a subjective quantitative assumption, however, Retained Austenite contents below 15% are hard to determine.

Magnet Inductive measurement is a volume measurement and needs to be calibrated to specific materials, heat treat and

geometries. The obtained results are usually higher than with XRD. No ability of profile measurements.

EBSD measurement. The results are somewhat similar to XRD with much higher efforts on preparation, operator and equipment.

XRD measurement is unproblematic with un-textured (isotropic) homogeneous steels. The presence of additional phases and reflections due to grain size, carbides or texture can cause disturbances and variances. However, the newest generation XRD machines can compensate for these obstacles and provide precise, repeatable, and fast measurements at a very competitive cost.

Acknowledgments

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APPENDIX

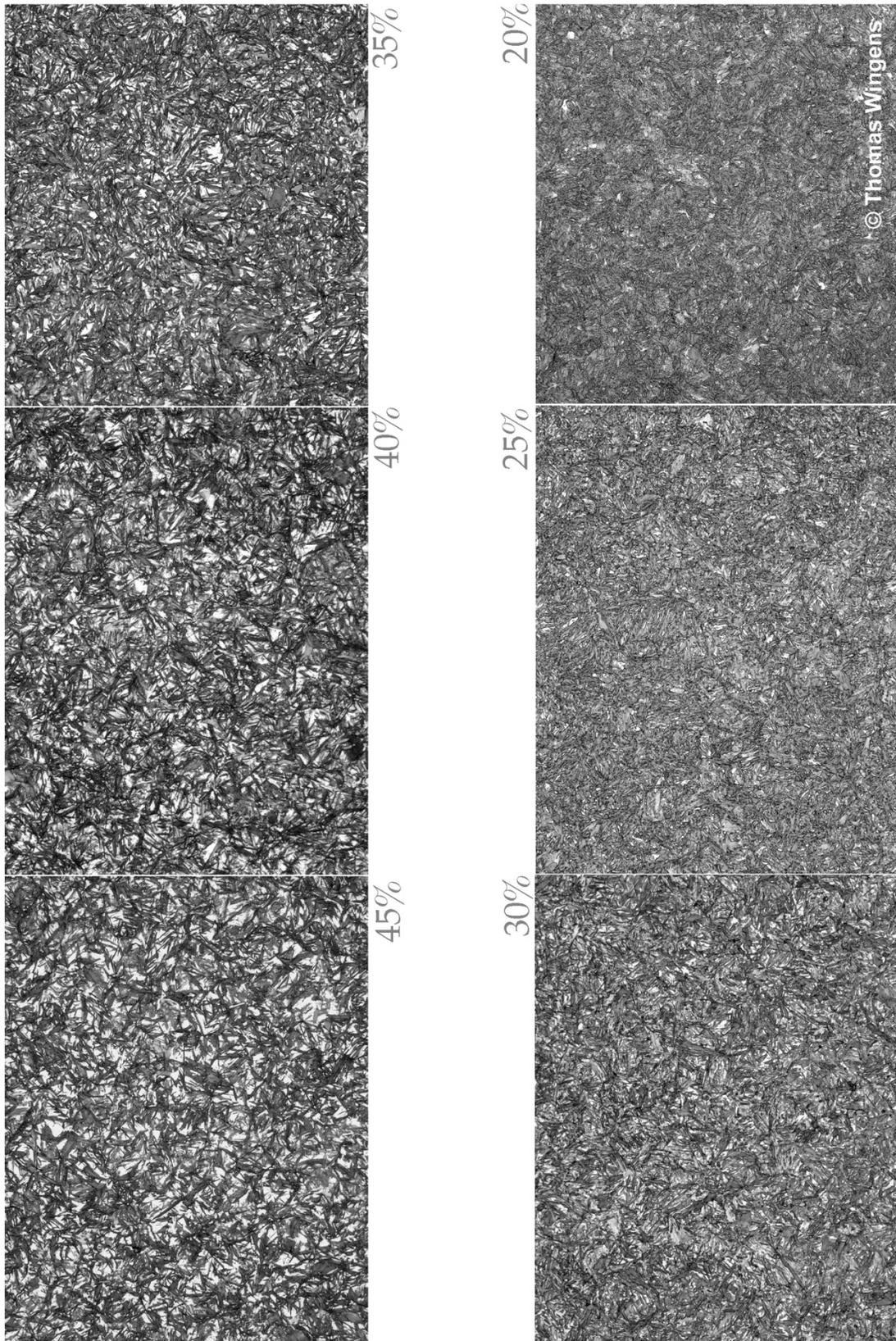


Figure 13: Metallographic picture standard for visual determination of Retained Austenite (500x magnification, 3% Nital etchant) (9)