

Predicative Hardness Testing for Production Control and Materials Design

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Abstract

In-situ high-temperature rebound hardness testing according to the EQUOTIP® principle is useful to study effects of secondary hardening on strength and thermal stability, e.g., of highly alloyed tool steels. In-line material characterization and in-production testing are now feasible and offer new possibilities for production control and materials design.

Introduction

Hardness tests have always been important in the metalworking industry and in materials research [1]. Specific mechanical properties for further processing or of defined final conditions are obtained in industrial production by specific heat treatments. Tool steels, e.g., are optimised for their applications by running specific heat treatment programs and by varying the alloying elements. Quantitative data for softening during high temperature annealing are required. These data can determine the integrity of the product and its possible lifetime.

Hardness testing is used amongst other methods to retrieve these corresponding information and to characterize these materials. Normally, hardness is tested at room temperature after the part has cooled down from annealing or tempering temperature, respectively. But here the part has already run through different metallurgical phases. Thus, the hardness test then hardly can be used to estimate the mechanical properties at elevated temperatures even after rapid cooling by quenching where additional effects can contribute like stress hardening and diffusionless phase transformation.

In-situ high-temperature hardness testing up to 700°C – as recently developed by the Materials Center Leoben, Austria [2] – is described to analyse precipitation and softening with time. An automatic rebound hardness tester according to the EQUOTIP® principle was used for this purpose. Energy dissipation can be reduced because of no reheating and in-line material characterization for in-production testing is feasible. Thus, testing and material sorting can be performed automatically during the heat treatment. Recent results obtained with this method on highly alloyed tool steels are discussed.

Rebound Hardness Testing according to the EQUOTIP® Principle

Since its introduction in 1975, rebound hardness testing according to the EQUOTIP® principle (also known as LEEB® hardness testing [3] – named after Dietmar Leeb, who with PROCEQ SA invented the method) turned out to become a very renowned method for production control in nearly all industries where metallic components are fabricated, heat treated, machined and maintained (see Fig. 1 for typical applications). Users appreciate especially its portability, ease of use and fast and accurate measurement over a long period of time. Due to its usefulness and the predicative measurements very frequently the whole series production is controlled by portable rebound hardness testers where now the need for an automated tester has grown up (Fig. 1d).

At the very beginning the new hardness value LEEB® (HL) was not known and conversion had to be supplied into traditional hardness values like Brinell (HB), Rockwell (HR) or Vickers (HV) where the users had already their experiences made to assess production quality. In the meantime more and more the genuine LEEB® hardness value according to the EQUOTIP® principle is accepted as decisive method to qualify production processes. So its significance and reliability for acceptance tests has been certified throughout many industries by written specifications.

Conversion into other scales has to be considered with care and is not anymore recommended on hardened steel in order to avoid problems related to changes in material

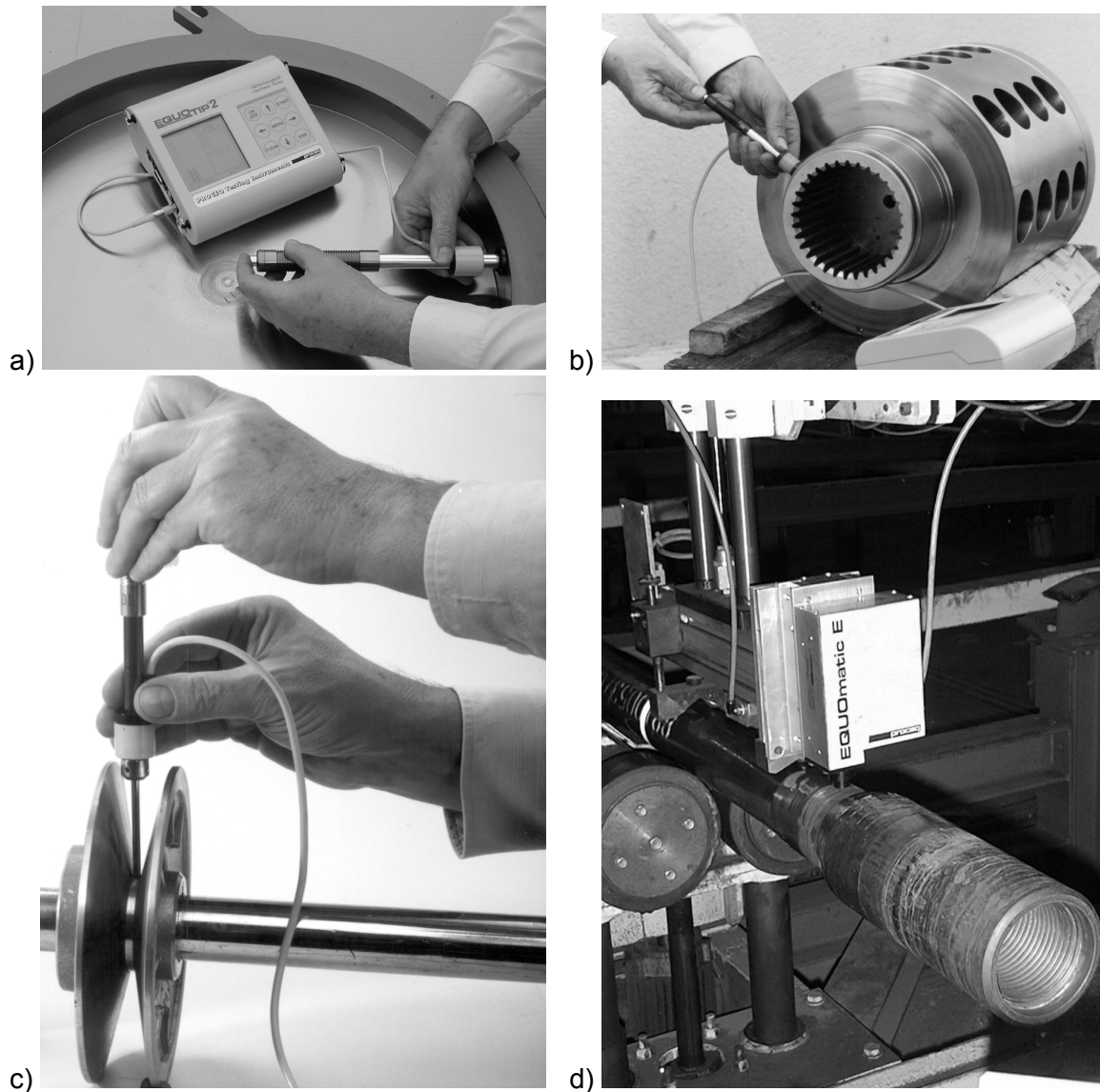


Fig. 1: Some typical applications for the rebound hardness tester according to the EQUOTIP® principle. a) The impact device G on heavy coarse grained, castings. b) The standard impact device D on machined parts. c) The impact device DL to access confined spaces. d) Automatic LEEB® hardness testing on drill rods.

properties due to handling (stress induced hardening for instance). The converted hardness value may divert from converted curves out of standard materials of similar grade. This effect is significant for all conversions between scales even for $HB \leftrightarrow HR$, $HV \leftrightarrow HR$, $HRx \leftrightarrow HRy$ etc. (x, y different indentation bodies or loads respectively). Therefore converted hardness values should be considered as informative estimation and not as acceptance values. The important exception is to have direct comparison of results between different hardness testing methods on the same material.

In 1999 the FORCE INSTITUTE, Denmark [5-7] has conducted a thorough investigation of several portable hardness testers on pressure vessel steels. They concluded that results gained with the rebound hardness test according to the EQUOTIP® principle should be accepted in general without conversion for above reasons. The widespread acceptance to the industries of the EQUOTIP® principle has led to it's standardization in 1996 (ASTM 956-96). Today EQUOTIP® is ready to be traceable according to NIST.

The Measurement Base HL

On the first view rebound hardness testing according to the EQUOTIP® principle is physically rather simple. There is a so-called impact body which is propelled by spring force against the surface of the test piece (Fig. 2). When the impact body hits the surface deformation takes place which in turn may result in an energy loss due to plastic deformation. This energy loss is calculated by speed measurement just before impact and then again directly thereafter. The ratio between rebound speed v_R and impact speed v_A is taken to calculate the hardness value HL (HL = hardness in LEEB® units):

$$HL = \frac{v_R}{v_A} \cdot 1000. \quad (1)$$

This means that a fully elastic rebound ($v_R = v_A$) would cause HL = 1000 or at the other hand HL will be lowered with decreasing material hardness. This procedure explains the naming of the EQUOTIP® principle where EQUO = Energy QUOtient and TIP = just TIP to trigger the measurement. The measurement procedure is shown in more detail in Fig. 2.

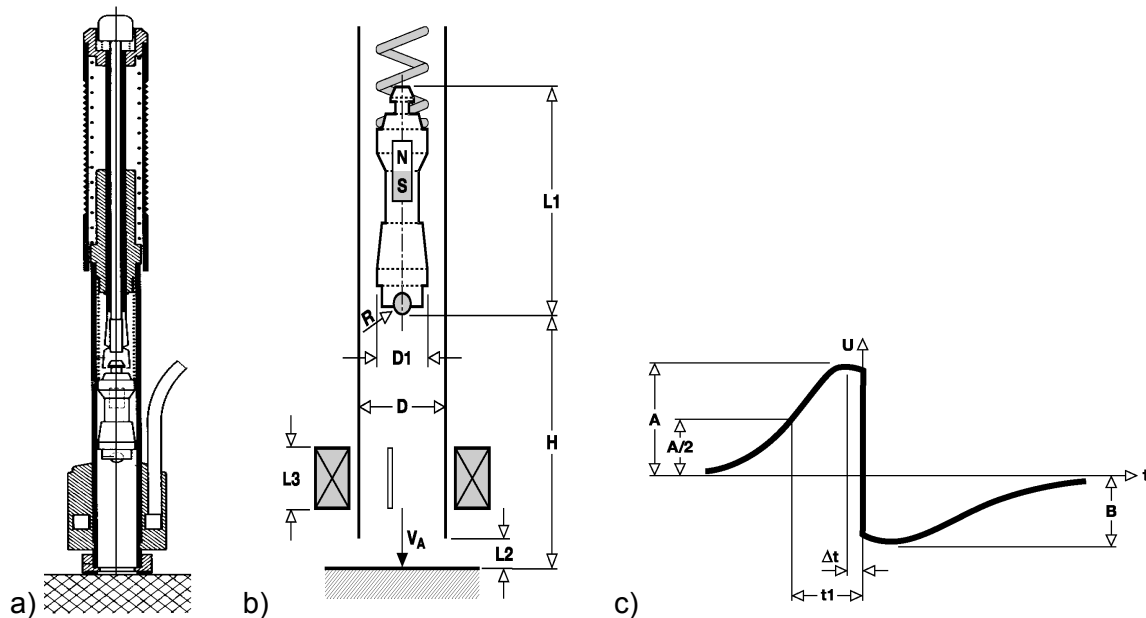


Fig. 2: Principle of standard single coil LEEB® hardness testers. a) Standard impact device D. b) Parameters defining the measurement base. c) Characteristic shape of induction signal generated within the single coil by the permanent magnet of the impact body.

For the standard single coil rebound hardness testing devices, a typical induction voltage curve is sketched in Fig. 2c where the shape of this curve is unique for all impact devices of this type. The impact and rebound velocities are assumed to be proportional to the extreme values A and B of the signal curve, which is a good approximation, if the device is constructed so, that the extremes are near the signal step caused by the impact. If they are too near, however, the reproducibility of the measurement suffers, because the signal is often slightly disturbed shortly after the impact. On the other hand when the distance becomes too long then internal friction may alter the measurement of rebound speed v_R . The width of the signal curve has some influence on the result, because it determines, how good the proportionality between minimum value B and rebound v_R velocity is.

Another important parameter determining the actual HL-value for a material of a given hardness is the impact energy, which follows from the impact velocity, the mass of the impact body, and its stiffness (which determines, how much energy the impact body absorbs). In order to reproduce the standard direction dependency, it is necessary to specify velocity and mass separately and to have a specific free flight length. Thus, the LEEB[®] testing method can be used for any direction. The results are, however, not completely independent on the impact angle. Each of the standard probes has its own characteristic direction dependency, which is determined by (i) the combination of the impact velocity and the free flight length of the impact body and (ii) the shape of the induction voltage signal, which is determined by the velocity vs. time curve on the one hand and by the characteristics of the sensor coil and the permanent magnet on the other hand. This means that the impact energy in general is the most important parameter for significance of HL-values for all rebound hardness testers working in units of the seven different standard impact devices; D, DC, E, D+15, DL, C, and G. Coil and permanent magnet are not explicitly specified. They have to be chosen in such a way, that the specified parameters of the induction voltage signal are fulfilled.

The impact devices D and E have become industry standards for general purpose applications since the first introduction of the D-device in 1975. The other types have been added with the time for applications with special requirements. Further details on the selection of the proper impact device and its use are found in the ASTM A956-00 Appendix X.1 [3] and the instruction manual of the manufacturer [4] where all the relevant specifications for impact devices D/DC, E, D+15, DL, C, and G are summarized.

It is well known that the HL-readings for a given specimen differ significantly, depending on the impact device type used. The main reasons for this are (i) different impact energies, (ii) different sizes and materials of the indenter and (iii) different stiffness of the impact bodies. Thus, the HL-value depends on geometry and material properties of the indenter, predominantly roundness, hardness and elasticity. Finally, the effect of deceleration by eddy currents may affect the result. So the tube material must be specified, too, as well as special precautions have to be taken to reduce eddy currents.

If the above precautions are not fulfilled unpredictable large deviations in HL-values may follow. A comparison of an out of series produced hardness tester and an original EQUOTIP[®] hardness tester is shown in Fig. 3. A wide hardness range was covered by using several standard reference blocks of different hardness. Quite often the measurements are good in the hardness range of standard reference blocks between 700 and 750 HL_D (index stands for the impact device D). But below and above the deviations are considerably large and the results by such testers have to be considered insignificant and erroneous.

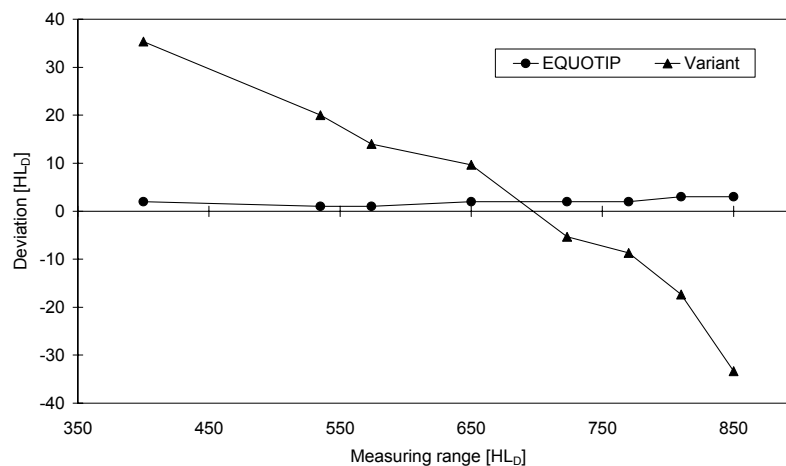


Fig. 3: Typical deviation of HL-values on standard hardness reference material given by EQUOTIP and variant.

Hot Hardness Testing on Tool Steels

This paragraph is a summary of a recent article [2] published by the MCL-Materials Center Leoben, Austria. All following graphs are courtesy of MCL.

A detailed understanding and quantitative description of the precipitation of secondary hardening carbides from the supersaturated martensite as well as the coarsening behaviour during tempering (annealing) are main goals for material scientists dealing with tool steels. The extreme fineness of the secondary hardening carbides makes it difficult or impossible to inspect them by direct microscopic techniques. Thus, indirect methods have to be employed to get additional information on the reactions and processes taking place. One of the most promising techniques is the in-situ hot hardness testing which can be used to draw conclusions on microstructural changes and avoids unknown effects on the hardness due to temperature changes during quenching. The authors concentrate on the softening behaviour of two tool steels in fully heat-treated condition.

The hot hardness testing device developed for the experiments took advantage of the EQUOTIP[®] hardness tester (impact device DL) from PROCEQ SA (Fig. 4). Common quasi-static hardness testing methods like Vickers or Rockwell were not suitable as the carbon of the diamond indenter dissolves by the hot steel and an optical determination of the indentation size is difficult and inaccurate. The EQUOTIP[®] indenter is made of cemented carbides or high speed steel and the testing occurs in a very short interaction time between indenter and sample. The HL_{DL} -value may be directly converted into Vickers hardness using the stored conversion table of the instrument. An estimation of hardness values from tensile tests on tool steel at elevated temperatures reveals satisfying accordance with the results from dynamic hardness testing.

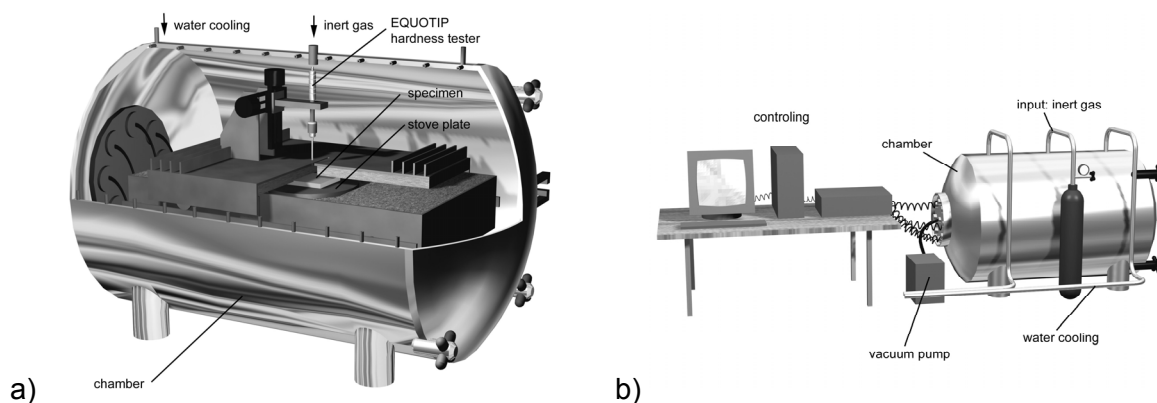


Fig. 4: Hot hardness tester (HHT); a) exploded view, b) total view.

In the semi-automatic test equipment the EQUOTIP[®] hardness tester device DL is mounted on a computer controlled three axis translation unit which allows hardness testing at predefined positions and times. To avoid undesired oxidation the sample is tested in a water cooled chamber which can be evacuated and subsequently flooded with argon gas. A high power heating plate is used to enable heating times less than 10 min to achieve 700°C. The hardness measurements can be carried out for a time up to 48 hours and up to 700°C.

Two types of austenitised and tempered tool steels were investigated; (a) HS6-5-2 (0.9 wt.% C-4.1 wt.% Cr-6.4 wt.% W-5 wt.% Mo-1.8 wt.% V, 10 min at 1200°C and 3 h at 615°C) and (b) X38CrMoV5-3 (0.38 wt.% C-5 wt.% Cr-2.8 wt.% Mo-0.65 wt.% V, 50 min at 1050°C, 1 h at 550°C and 2 h at 610°C). HS6-5-2 had a final hardness of 60-62 HRC and X38CrMoV5-3 of 50-52 HRC. Since dynamic hardness testing strongly depends on the mass, e.g., if the specimens are too small, specimens with dimensions of 100 x 100 x 20 mm³ were used to avoid mass effects. The effect of the annealing time and temperature on

the hot hardness is shown in Fig. 5 for both samples. Annealing temperatures of 600 and 650°C were chosen. The hardness changes are followed over 24 hours (1440 min).

A comparison of the hardness curves in Fig. 5 reveals the materials influence. The high speed steel HS6-5-2 shows a significantly higher hardness than the hot working tool steel X38CrMoV5-3, which is caused by the higher amount of secondary hardening carbides in the high speed steel. The lower hardness of the 650°C curve at the beginning of the diagram is mainly caused by the higher temperature, but also the heating time necessary to achieve the higher temperature causes additional softening before starting the experiment. The hardness curves reveal the dominant role of the annealing temperatures on the softening rate. The hardness loss seems to be about one order of magnitude faster in case of the higher annealing temperature (650°C) than for the lower annealing temperature (600°C).

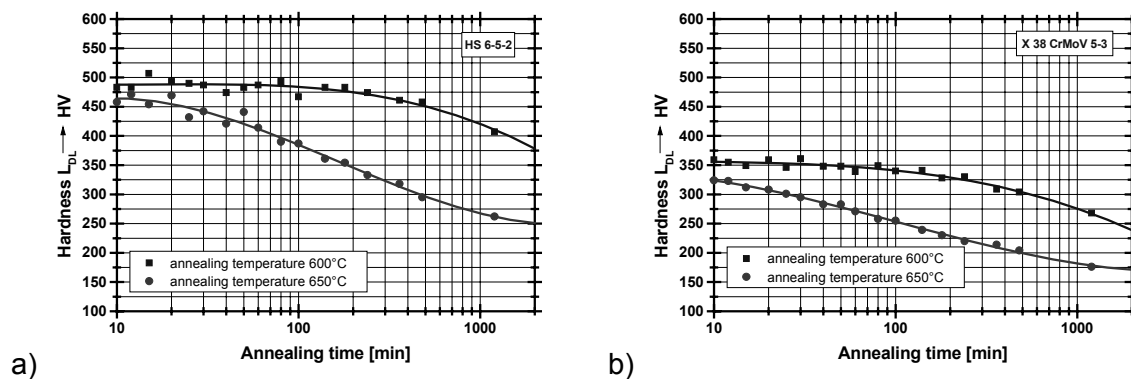


Fig. 5: Annealing curves a) of the high speed steel HS6-5-2 and b) of the hot working tool steel X38CrMoV5-3

Hot hardness testing according to the EQUOTIP[®] principle has been proven to be significant within its unique measurement base HL when above requirements for the test system are fulfilled. It shows good potential for materials design and in-process control of series production during heat treatment. Further standardization work is necessary to achieve the required quality for all variants currently on the market [5-7].

References

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