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ABOUT THE CHOICE OF TUNGSTEN CARBIDE INDENTER TO DETERMINE MECHANICAL PROPERTIES OF SUPERALLOYS BY USING HIGH-TEMPERATURE MICROHARDNESS TESTER

Bruno Passilly*, Amélie Kardache

Université Paris Saclay, ONERA, DMAS, 29 Avenue de la division Leclerc, 92320 Châtillon, France

* corresponding author: bruno.passilly@onera.fr

Abstract. In the aeronautical field, materials are used in severe environmental conditions (temperature, atmosphere), particularly in engine applications. In order to qualify mechanical properties of new composition Ni-based superalloys, ONERA performs Vickers hardness tests from room temperature up to 750 °C close to operating conditions. This method consists in applying a pyramidal tip onto the specimen to characterize hardness and mechanical resistance of the material. This simple method appears to be faster than other methods using classical hot tensile or bending tests.

Nevertheless, the choice of the indenter tip for high-temperature experiments is crucial. Tungsten carbide tip is used for characterizing Ni-based superalloys. Electron microscopy and X-ray analysis are presented and discussed on new and used tungsten carbide tips. A simple experimental method to control the evolution of the indenter before and after using it in the high-temperature hardness test is explained.

Evolution of hardness and mechanical resistance versus temperature by using Tabor relationship on a Nickel-based superalloy sample is compared to evolution of mechanical resistance values determined by classical high temperature tensile tests. A good agreement is found between these two methods with WC indenter. These hardness measurements could be carried out up to 1 000 °C if indenter is still available to characterize layers, coatings, composite materials, additive manufacturing materials or gradient properties materials.

Keywords: High temperature, hardness, superalloy, mechanical resistance, indenter, Ni-based superalloy.

1. Introduction

The development of the aerospace industry during the last century led to elaborate new materials to increase aircraft performances. Studying new Nickel-based superalloys demands a better knowledge of mechanical characteristics of each new superalloy composition [1].

Conventional high-temperature mechanical tests such as tensile and bending require a large quantity of superalloy. The equipment is heavy and expensive, and a longtime preparation of a lot of specimens as well as long test periods are required to investigate mechanical behavior of only one new superalloy for each tested temperature.

In order to increase the quantity of results on the behavior of superalloys in severe thermal conditions, it is essential to test each new composition by developing a high temperature micromechanical test. A lot of nanoindentation means have been developed over the past twenty years for room temperature usage [2] and more recently at high temperature in a limited scale of loading [3]. In order to characterize new superalloys for turbomachinery parts, simple hardness measurements are sufficient to determine hardness and mechanical resistance of a small quantity of the specimen without having to apply a maximum load less than 1 N and to measure displacement of the indenter. Preparation of specimens is easy and the test period is short. The evolution of hardness and mechanical resistance of Ni-based superalloy versus temperature up to 750 °C are presented and discussed by using the WC indenter.

2. High-temperature hardness tester and indenters

2.1. High-temperature hardness tester

The high-temperature hardness device shown on Figure 1 and 2 is a prototype developed by ONERA [4]. The maximum load P that could be applied by the indenter on the sample is up to 30 N. The maximum temperature that could be reached by the furnaces is about 1 000 °C. Working atmosphere is composed of argon with 3 % vol of hydrogen under a pressure of working 0.5 bars. This special gas contains less than 0.2 ppm.mole of oxygen.

The tip and the specimen are separately heated by cylindrical furnaces to minimize thermal gradient between. The hardness tester has stable and efficient thermal regulations that allow fast heating and little overshoot [5]. A thermocouple is fixed in the ovens near the sample and near the indenter to control and regulate the temperature during tests. The load applied to the sample is measured by load sensor

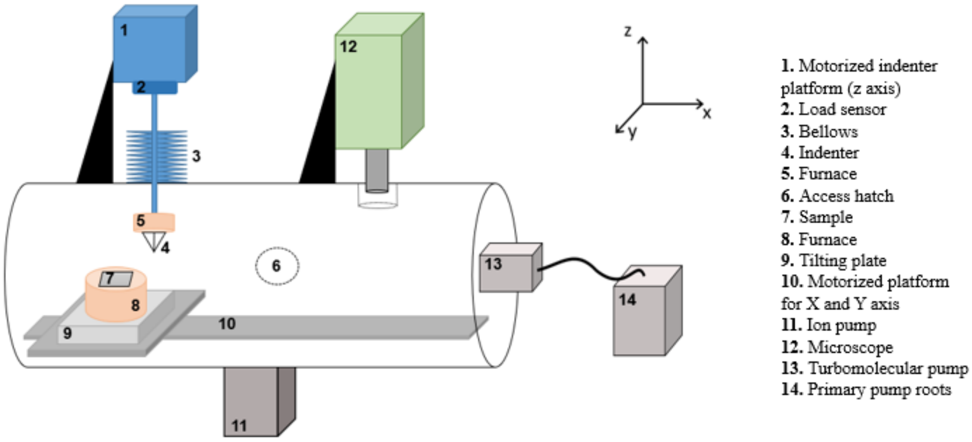

Figure 1. Diagram of the main components of the hot indentation apparatus.

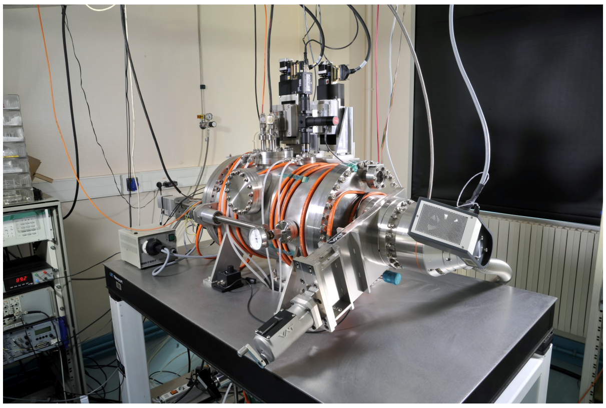

Figure 2. High-temperature microhardness prototype developed by ONERA.

(LCM201-100 N, Omega) which is located outside the vacuum chamber. The movement of the indenter is controlled by a motorized table which compresses a bellow when the table is moving to indent the sample. A second motorized displacement table supports the optical microscope outside the vacuum chamber to focus indentation zones and to measure the imprints. A cooled porthole is installed between the sample and the indenter to avoid any heating of the lenses.

2.2. Indenter choice for high temperature tests

2.2.1. Indenter materials principles

During an indentation test, the tip must be deformed only elastically and must have a high hardness to be less deformable than the tested material. According to J. M. Wheeler and J. Michler [6–8], indenter must be 20 % harder than material being tested, otherwise the tip will be eroded quickly and will break. To avoid this breakage problem, the tip must have a hardness significantly greater by 100 % or more than that of the material (Figure 3). For hot indentation, it is then necessary to know the hardness of the indenter at high-temperature test.

The choice of the material composing the tip must result necessarily from a compromise between a large number of criteria. Indenter material must have good geometric stability at the test temperature. It must have good chemical stability without phase change or chemical reactions with environment or reaction with the tested material. It must keep high hardness and good thermal conductivity at high-temperature. And finally, indenter must be easy to manufacture and to install on the setup.

Another way to avoid damages on the indenter during indentation tests at high temperature is to minimize time of contact of the tip with the specimen. Tiphene shows typical cycle of loading and unloading in less than 1 second [9].

2.2.2. Indenter holders

Brazing is a permanent heterogeneous assembly process that achieves metallic connection between the two joined parts. To assemble indenter and holder, a filler metal is employed. When the melting temperature of the filler metal is reached, the joint is formed by capillarity. Hard brazing is recommended to reach 600 to 1 000 °C. Nevertheless, it is recommended to use theses assemblies at a lower temperature than half the melting temperature of the filler metal. Ultra-High Temperature (UHT) vacuum brazing using molybdenum with a melting temperature of around 1 450 °C allows a working temperature of 750 °C. This type of solder was used on a sapphire tip by Minnert [10] and had no problem rising to 1 100 °C. Furthermore, during the test, if the melting temperature of the filler metal was reached, the tip would move and then it would be oriented incorrectly for the next tests.

Another way consists in blocking the indenter on a support by using a screw as it can be seen in Figure 3. The advantages of this process are that the support is reusable and can resist up to 1 400 °C. However, this way is more interesting if the indenter is in one piece, without brazing. This method requires more material to make an indenter and manufacturers using this

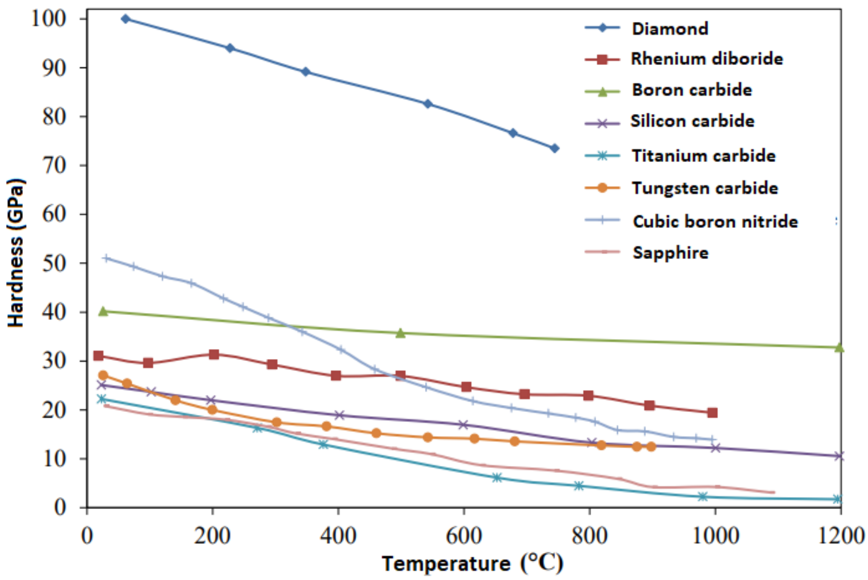

FIGURE 3. Hardness of different materials versus temperature [6].

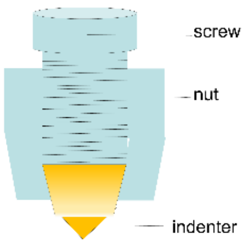

FIGURE 4. Sample holder with screwing element.

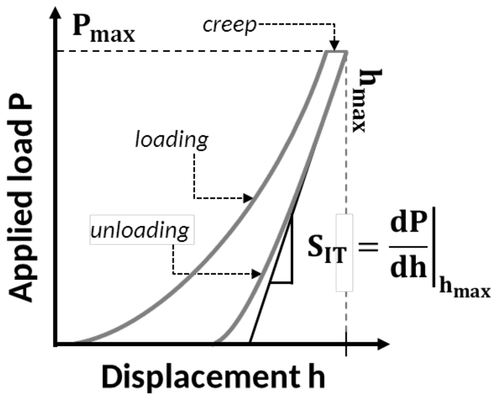

FIGURE 5. Schematic graph presenting the multiple phases of the loading cycle during indentation tests [2].

technique are very few in the world. To consolidate everything and ensure that the screw will not unscrew, a ceramic cement is used to secure the screw.

On the high temperature hardness tester, WC tip is brazed on the holder, and this assembly is screwed on the instrument like in Figure 4.

3. Results on superalloy

3.1. Generalities about superalloy

Superalloys suited for single crystal casting derived from nickel-based superalloys. The alloys' designers succeeded in optimizing the mechanical properties of these alloys by introducing large amounts of refractory alloying elements such as W, Ta, Mo. Caron et al. [1] described the evolution of the chemistry of this class of alloys by focusing on advantages and drawbacks resulting from these chemistry changes.

3.2. Hardness relationship

During microhardness testing, the applied load is measured as a function of the displacement of the indenter in the material. This produces an indentation curve like the one shown in Figure 5.

In metalworking industry, Vickers indenter (a square-based pyramid tip) is preferentially used. Moreover, this shape of indenter is used on a commercial Vickers hardness tester (Wilson VH3100, Buehler) in order to compare the hardness value with our device.

As there is no displacement sensor on the hot hardness tester, Vickers hardness is calculated from the imprint left in the material and not from the load-unload curve. Microhardness is then calculated with the following Equation (1):

$$H_v = 1.854 \times \frac{F_{max}}{d^2} \tag{1}$$

In this relationship, F_{max} represents the maximum applied load during the indentation, and d the average between the two diagonals of the Vickers pyramidal imprint.

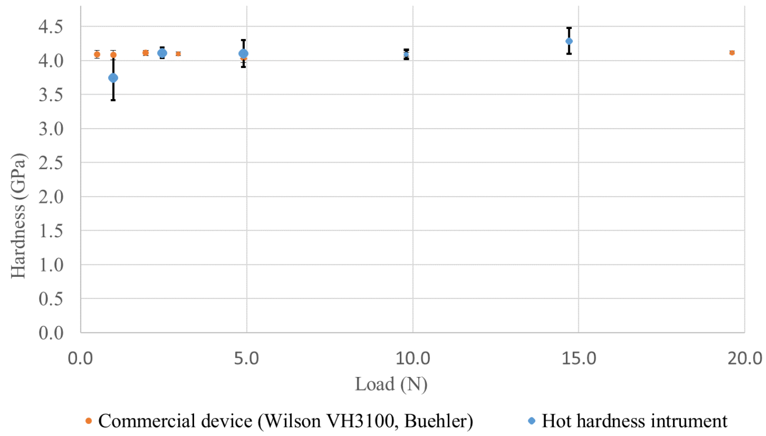

FIGURE 6. Hardness of superalloy versus applied load on a commercial tester and hot-hardness tester.

3.3. HARDNESS VERSUS APPLIED LOAD ON NI-BASED SUPERALLOY

The high-temperature hardness tester is calibrated at room temperature on known metal alloy stainless steel 316L showing a low dispersion of the measured values of hardness which were compared to those obtained on commercial room temperature Vickers hardness tester (Wilson VH3100, Buehler) [4]. Figure 6 represents evolution of hardness versus applied load. An average of 5 measures for each applied load is calculated and the standard deviation is represented.

3.4. HARDNESS VERSUS TEMPERATURE ON NI-BASED SUPERALLOY

For each chosen temperature, the hardness measurements are collected from hot-hardness test on a Ni-based superalloy (Figure 7). An average of 5 measurements for each temperature is calculated, and standard deviation is presented on Figure 8. Hardness is decreasing slowly from 20 °C up to 600 °C, and is more important after 600 °C.

3.5. MECHANICAL RESISTANCE

Tabor's law [11] is used to express the mechanical resistance R_m from the hot Vickers microhardness result H_v, following Equation (3):

$$R_m \approx \frac{H_v}{3} \qquad (2)$$

It is important to note that the coefficient of proportionality 3 set up by Tabor is an average obtained after a study on different metallic materials (steel, aluminum, copper, etc.) [11, 12]. After normalization of the mechanical resistance values calculated from hardness and tensile tests on the same Ni-based superalloy, the values obtained experimentally follow the same decrease at the same temperature with a lower percentage value in the case of hardness experimental data (Figure 9). This result allows us to affirm that the used indenter is operational up to 750 °C.

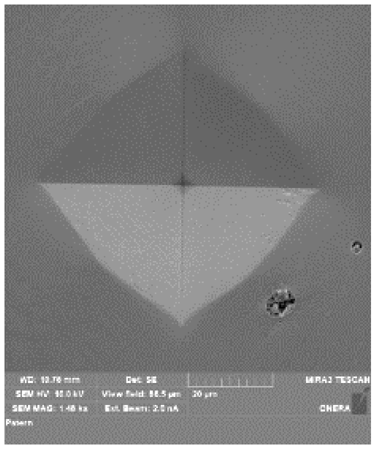

FIGURE 7. Typical imprint of a Vickers test on Ni-based superalloy under a load of 10 N.

3.6. SCANNING ELECTRON MICROSCOPY ON TUNGSTEN CARBIDE INDENTER

In order to observe its surface conditions, new and used tungsten carbide tips are analyzed by using Electron Microscope. The new Vickers tungsten carbide tip has a relatively smooth surface, but the presence of porosity is noted on Figure 10. Figure 11 shows the WC indenter after 20 hours of heating at 750 °C and 2 hours of mechanical stress between the tip and the superalloy. The indenter is slightly blunt and appears to be slightly damaged, with few contaminations of the tip.

Tungsten carbide tips have good mechanical resistance during indentation tests. A qualitative analysis of the indenter is carried out on an area located in the region of contact between the indenter and the tested material, in the periphery of the indented zone and in the unstressed zone. The presence of aluminum, silicon and nickel is detected only in the region in contact with the material. Some elements present in the alloy were stuck in the porosities of the tip. Tip cleaning might be necessary to avoid this contamination [13].

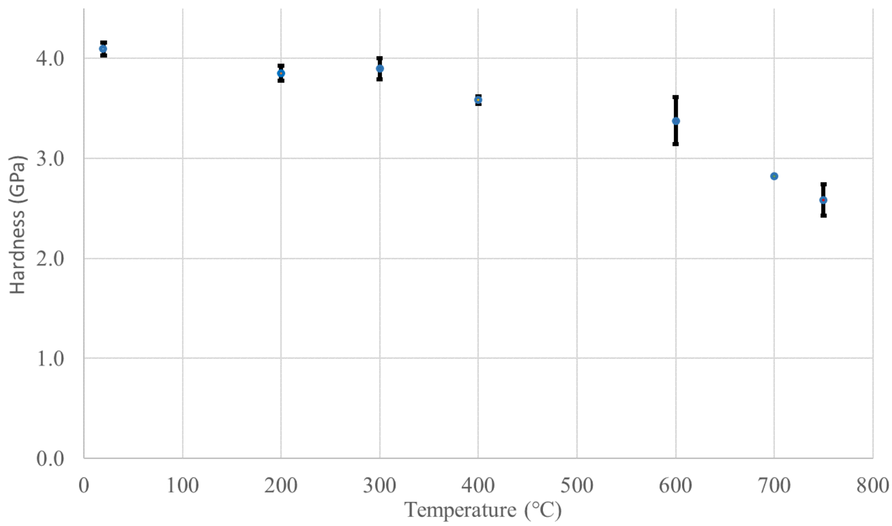

Figure 8. Hardness versus temperature of Ni-based superalloy on hot hardness tester.

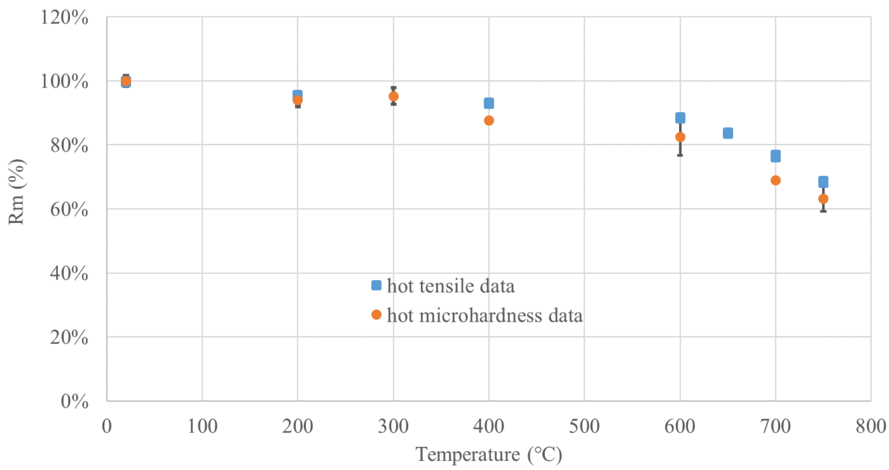

Figure 9. Normalization of R_m as a function of temperature for Ni base superalloy.

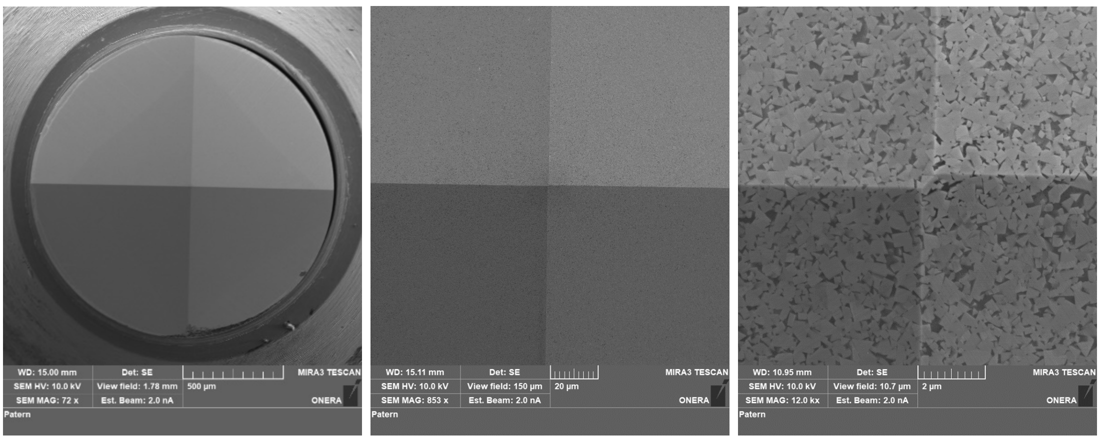

Figure 10. SEM image of new Vickers WC tip at different scales.

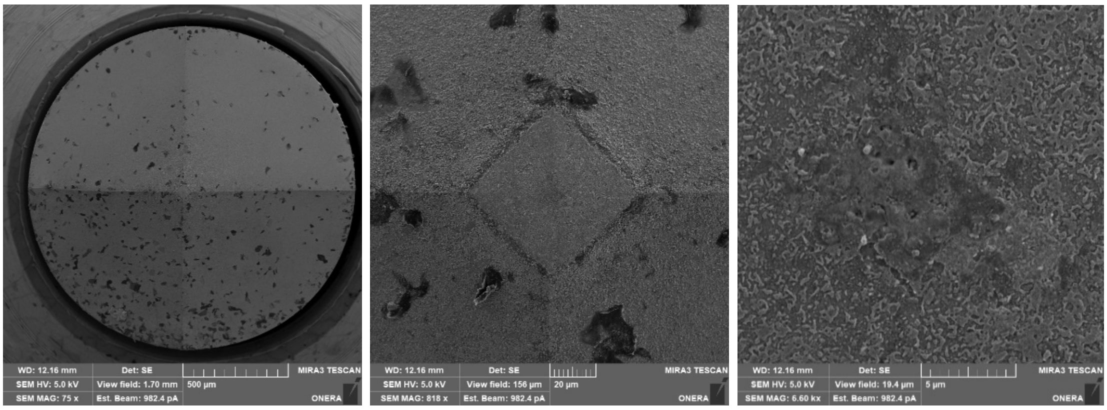

FIGURE 11. SEM image of used tungsten carbide indenter at different scales.

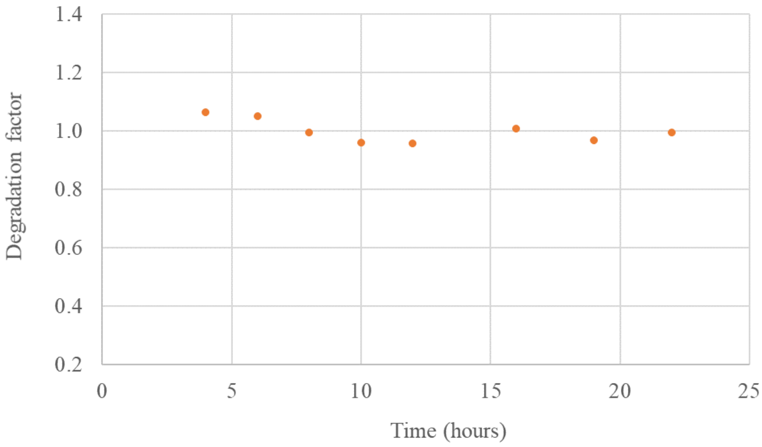

FIGURE 12. Corrective factor versus time of heating for WC tip at 750 °C.

3.7. Life duration of the WC indenter

Indenter could react chemically with the materials to be tested and could be sensitive to different environmental factors such as temperature and working atmosphere. Tip could become fragile, develop cracks, or deteriorate, then the indentation results would be affected. In order to notice a possible degradation of the tip as the tests progress, the evolution of a degradation factor K is measured :

$$K = \frac{Hv_{ht}}{Hv_{rt}} \tag{3}$$

Hv_{rt} is the hardness measured on a room temperature conventional microhardness tester (Wilson VH3100, Buehler). After each series of test at 750 °C on the high temperature hardness tester, hardness Hv_{ht} is measured at room temperature.

Consequently, tip degradation can easily be represented as a function of the time spent by the tip at high temperature. Figure 12 shows the evolution of degradation coefficient K versus time of heating. After 22 hours at 750 °C, variation of K is less than 5 %. This result correlated with the SEM images demonstrates that the tungsten carbide tip is still usable to provide hot hardness results.

The tungsten carbide tip resists mechanically and chemically to high-temperature micro-hardness testing on Nickel-based superalloys. The evolution of this degradation factor is a good indicator to determine precisely the start of tip deterioration and therefore the usefulness of replacing it with a new one.

Tungsten carbide WC appears to react moderately with oxygen and can react slightly with certain transition metals. This indenter is chemically stable under high-temperature conditions and under inert gas atmosphere.

4. Conclusions

Hot-hardness tester prototype can perform a large number of high-temperature tests in just a few hours. In addition, this type of test is non-destructive and requires little quantity of materials and simple preparation. Hardness is therefore a very interesting characterization technique since it is useful to determine certain local mechanical properties of materials.

The characterization of Nickel-based superalloys is performed for a load of 10 N and up to a temperature of 750 °C with WC indenter. This test allows us to determine the evolution of the hardness of a Ni-based superalloy or mechanical resistance as a function of temperature by using Tabor's law. When these values are normalized, the variation of the percentage of R_m versus temperature, obtained in hardness, is very close to the R_m obtained in traction and follows the same curve shape with a pronounced decrease after 600 °C.

Unlike tests at room temperature with the systematic use of diamond, it is necessary to consider an indenter-material pair for each series of tests on material. Indeed, the predominance of chemical reactions between the material tested and the indenter does not currently make it possible to find a universal indenter for high-temperature tests.

Monitoring the health of the indenter is essential to avoid experimental bias leading to erroneous results. For this, developing new high-temperature indentation devices under a scanning electron microscope is promising. Indeed, the combination of these two techniques will allow the qualitative observation of the formation of imprints and the interaction between the tip and the sample. This interaction highlighted in this work is certainly harmful for tests carried out at high load, and risks being even more harmful for tests in the nanoindentation range.

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