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HOT HARDNESS MEASUREMENTS ON MATERIALS UP TO 600 °C DURING THE FIRST HOUR OF USING

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Abstract. In the aeronautical field, materials are used in severe environmental conditions (temperature, atmosphere, exposure time ...), particularly for engine applications. In order to characterize the use of these materials in the evaluation of their properties, it is necessary to carry out tests in conditions close to their operating environment. Hot hardness is a simple method which can be applied on many different materials such as oxidized layers, coatings, composite materials, brazing cords, additive manufacturing materials. ONERA is developing micromechanical characterization means to carry out Vickers microhardness tests from room temperature up to 600 °C. In principle, a pyramidal punch is applied on the surface of a material and the applied load is continuously measured during indenter's moving in the material. The material is tested locally under conditions close to the actual conditions of employment. The goal of this research is to improve microindentation in order to achieve temperature test campaigns up to 600 °C under a controlled atmosphere of argon and to validate a method to produce a series of results during the first hour of using up to 600 °C. Stainless material is studied to compare the evolution of its hot hardness properties versus different parameters such as load, holding time at the maximum load, atmosphere, and thermal duration. A discussion about these measurements and the technical limits of hot hardness technology is presented.

Keywords: Hot hardness, microindentation, stainless steel, temperature.

1. Introduction

In aeronautic applications, temperatures can reach over 1000 °C in work condition. The used materials have to withstand stresses, strains as well as temperature cycles which can lead to the damage of the part. Therefore, material characterization plays an important role in the part design. Microindentation and hardness tests present many advantages compared to classical characterization methods such as flexural or tensile test: a simple geometry, little material quantity needed, automated data collection and analysis. It can be performed on substrate, thin films or through several layers too [1, 2]. When temperature is increased, it has an impact on the structure and the properties of materials. Measuring hardness at high temperature gives important insight of the thermophysical properties of the material. Research on micro and nanoindentation at high temperature has increased for the past two decades and many issues remain [3–5]. Techniques have been developed to reduce induces thermal dilatation, thermal drift and oxidation which affect the quality of the results. The goal is to validate a method to investigate the changes in hardness versus temperature under the first hour of heating and to avoid thermal drift and oxidation of the indenter or the sample [6–8].

2. Hot hardness set-up

2.1. Presentation of the experimental set-up

The microindentation machine is composed of the chamber which comprises the sample holder, an optical microscope, the indenter, the indentation station, 2 motorized (x,y) tables to move the sample from the chamber's opening to the microscope or indentation station, 1 motorized table to move the indenter to the sample and 1 motorized table to move the microscope for focusing (Figure 1 and Figure 2). A primary pump and a turbo pump are necessary to empty the vacuum chamber. A gas tap is required to introduce Argon + Hydrogen in the chamber. A water cooling system regulates the temperature of the measurements. A computer and a developed software on @LabVIEW are needed to manage all the movements of the tables and to acquire all the datas versus time of the sensors like thermocouples, load sensor, displacement coordinates of the tables. The following Table 1 sums up the characteristics of the machine.

T_{max}	800∼°C
F_{max}	3N
Atmosphere	Ar+3%H2
Indenter's shape	Vickers
Indenter's material	Diamond

Table 1. Characteristics of high temperature microhardness set-up

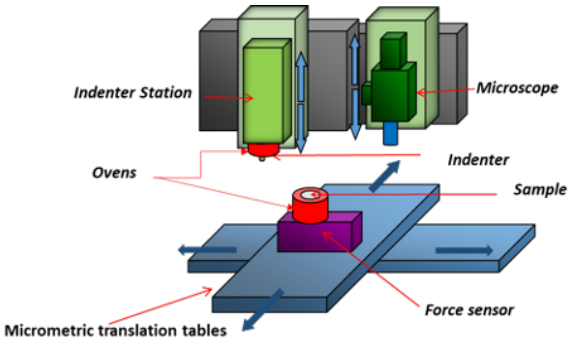

FIGURE 1. High temperature microhardness synoptic

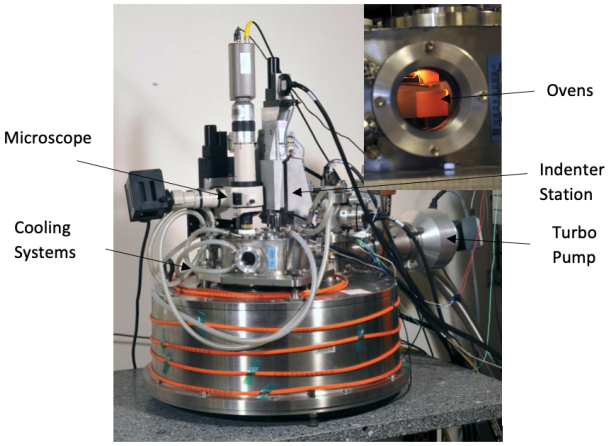

FIGURE 2. High temperature microhardness set-up

The two ovens are composed of the heating part, which is made of a platinum coil which is winded around an hollow cylindrical ceramic part. The sample is placed inside the ceramic part of the first oven, and the indenter is placed in the second oven. A current goes through the coil and heated up because of Joule's effect. A thermocouple sensor measures ovens temperature and the controller fixes the working temperature, the slope of the rise ramps temperature and power. As the experiments are carried out at high temperatures, the oxidation of the sample's surface has to be carefully avoided. In order to work under argon and hydrogen gas, vacuum of the chamber first needs to be done to reach a residual pressure of 5.10^{-5} mbar minimum. The gas tap is opened and the chamber's door closed at the same time and the working pressure near from the atmospheric pressure is reached in 3 minutes. The primary pump is turned off once the turbo pump no long turns.

2.2. METHOD

The experiments are performed under argon and hydrogen gas at atmospheric pressure. The imprint calibration and the force sensor calibration are made prior to any experiments. The duration of heating must be optimized and controlled for different temperatures. For each oven, the study is performed at 400 °C, 500 °C and 600 °C. The investigated slopes are 15 °C/min, 25 °C/min and 50 °C/min. From each temperature, the time to reach the temperature as well as the overshoot (maximum temperature reached by the oven) is determined. The delay between the oven and the sample's surface is also determined. All of these temperature measurements are tested on the same material inox 316L-1 (Table 2).

Sample ref. num.	Temp. test	Study Remarks
316L_1	20, 200,400, 500,600	Temperature parameters
316L_2	20	Applied load and hardness
316L_3	600	Time duration
316L_4	400	Hardness
316L_5	500	Hardness
316L_6	600	Hardness

TABLE 2. Samples reference

2.3. TEMPERATURE PROFILE

Figure 3A shows the temperature profile of the sample's oven for different ramps and temperatures. It can be seen that the increase in temperature is steady and the set temperature is easily reached: the ovens are well regulated. However, an overshoot is observed. Even if it cannot be avoided, it needs to be as small as possible in order not to overheat the sample. To highlight this phenomenon, the curve is zoomed in (Figure 3B). The overshoot of the oven is lower than 2 °C. Therefore, 50 °C/min ramp is chosen as settings for all the hardness tests as it allows a faster heating with a reasonable overshoot.

2.4. TEMPERATURE DELAY AND TEMPERATURE DEVIATION

As the temperature of interest is the temperature of the sample, the following experiment focuses on the temperature delay and the temperature deviation of the sample's surface to the set temperature. A thermocouple sensor is placed just at the surface of the sample. The sample is placed underneath the indenter and the surface temperature is measured. The investigated temperature is 400 °C at 50 °C/min. Then, the distance between the ovens is decreased to see its influence on the surface temperature. There is 37s seconds delay between the oven's temperature and the sample's one (Figure 4A). The sample reaches a constant temperature of 400 °C with an overshoot which is less than 2 °C. However, the temperature difference is 6 °C and the temperature does not seem to increase.

The temperature deviation between the set temperature and the surface temperature is plotted against the distance between the two ovens (sample and indenter). For all temperatures, the deviation decrease may be observed when the ovens are brought close

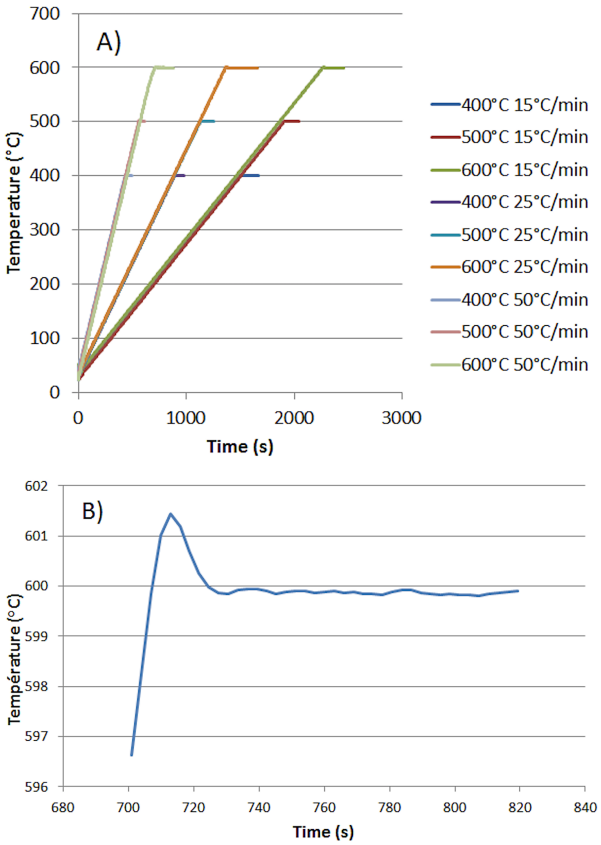

Figure 3. Temperature ramps for 400, 500, 600 °C set temperature and 15, 25, 50 °C/min ramp (b) overshoot example at 600 °C and 50 °C/min ramp

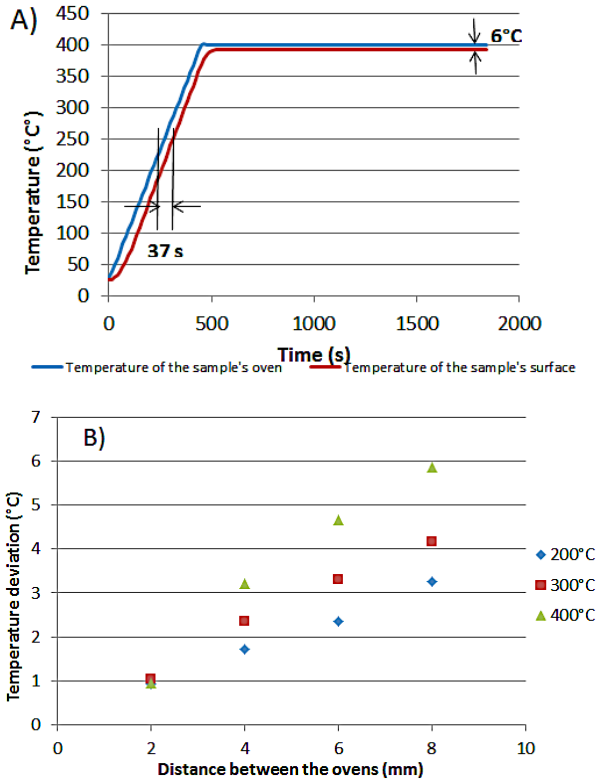

Figure 4. (a) Temperature difference between the thermocouple of regulation of the oven and the temperature of the sample (b) Temperature deviation as function of the distance between the two ovens

together. At 2 millimeters the deviation reaches only 1 °C difference. For each temperature test, the results can be extrapolated and it can be considered that there is no temperature deviation at contact.

3. Hot hardness results on stainless steel sample

3.1. Samples preparation

The material studied is a stainless steel 316L [9]. The material is cut in a parallelepiped shape 6 mm × 5 mm × 10 mm. Sample surface (10 mm × 6 mm side) polishing is performed with polishing papers with decreasing grain sizes from P800 to P4000. Diamond slurry is applied on velvet sheets with grain sizes of 3 µm, 1 µm and 1/4 µm. Between each step, the sample is cleaned in an ultrasonic bath of water and dried. An optical microscope is used to check the quality of the surface of the sample and to avoid scratches. The last step consists in cleaning the sample in an ultrasonic bath in acetone for 2 minutes and dried. For this study, 5 samples are prepared, sample 1 is used only to develop temperature profile before the study.

3.2. Measurement of hardness

The following protocol is applied: an imprint is done for a recorded load. Each imprint is separated by 100 µm of the following imprint. The imprints are measured using an electronic caliper square on the printed photo of the screen. For a Vickers imprint, each diagonal of the pyramidal imprint is measured separately. Hardness is then calculated by using Equation (1):

$$H_{\text{Vickers}} = \frac{P}{A_{\text{Vickers}}} = \frac{2.P \cdot \sin\frac{\psi}{2}}{d^2} = 1.854.\frac{P}{d^2}. \quad (1)$$

With P the load, d the mean of the measured diagonals of the imprint and ψ is equals to 136 °.

3.3. Study of the effect of the applied load and duration time

A series of 5 indents is performed for each following load 50, 100, 150 and 200 grams. Equation (1) is used to calculate the hardness. If the mean values are considered, the hardness values are constant as the load is increased for a given temperature of 20 °C. The lowest standard deviation is obtained for a load of 150 grams, which will be the load to be applied for all the other tests.

The effect of the duration of time at the maximum load is also investigated. A 150 g load and a temperature of 600 °C are chosen. The same protocol as the load effect is applied: 5 imprints are done for each load, separated by 100 µm. The results of hardness at 5, 180 and 360 seconds duration time at the

maximum load are constant which show that no effect of duration time at maximum load is occurring. A 5 seconds duration time is chosen for all the following experiments.

3.4. Thermal history and changes in hardness

The ovens are heated to the set temperature, then the contact point and indentations are made for one hour. The imprints are observed at the optical microscope. The temperature is set to the temperature test, the load used is 150 g and the duration time at maximum load is 5 seconds. For each temperature a new sample of stainless steel 316L is taken to have the same thermal history for each series of points (see Table 2). The Figure 5 shows an hardness mean of 3.6 GPa at 20 °C which decreases at 200 °C to reach 2.5 GPa and is stable for a higher temperature up to 600 °C. In order to investigate the possible oxidation layer, a series at 20 °C was performed after each series at high temperature were done. The values of the second series at 20 °C matches the first one. Thus, the sample's oxidation is avoided.

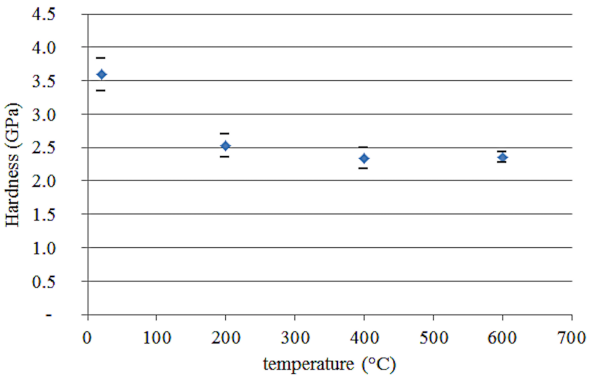

Figure 5. Variation of hardness on stainless steel up to 600 °C

The final goal is to validate this method to measure the changes in hardness in less than one hour at 600 °C. Figure 6 represents constant value of hardness over time once 600 °C is reached. The mean value of the hardness is 2.4 ± 0.08 GPa. At 20 °C there is no more evolution of hardness than at 600 °C.

4. Conclusions

To conclude, the study shows very stable and efficient thermal regulations that allow fast heating and little overshoot which are not reach on conventional microhardness apparatus. Oxidation problems are avoided by working under argon atmosphere. Changing samples for each temperature is a good method to compare the results with the same thermal history. The tested method seems promising and higher temperatures (up to 1000 °C) under ultra-high vacuum or argon atmosphere should be developed to characterize materials which are used under more severe conditions.

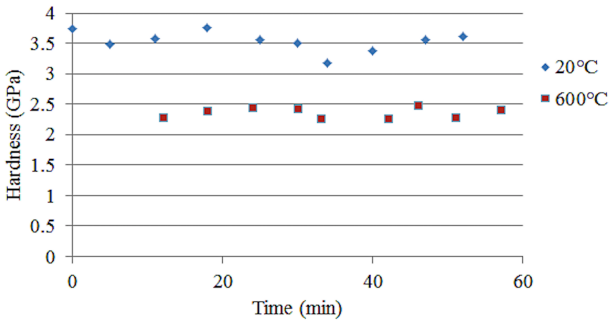

Figure 6. Validation of the method at 600 °C and 150 g load on a new sample

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