Surface and Subsurface Metallic Inclusions Detected using Hot Tip Thermoelectric Power Measurements

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Abstract. In this paper, we present a relatively new technique that can give informations on surface and subsurface metallic defects, i.e. the Hot Tip ThermoElectric Power (HTTEP) measurement. We show that depending on the size of the tip, different metallic inclusions in size and depth can be detected. We develop a numerical simulation based on the resolution of the transport equation in order to test the viability of the technique. We discuss the influence of the nature, the size and the depth of the inclusions on the measured TEP. The simulations results are compared with HTTEP measurement obtained on artificial metallic defects in samples. We show that by choosing judiciously the size of the tip, surface and subsurface metallic inclusions can be detected.

1. Introduction

Most metallic materials contain inclusions which can be either metallic or non-metallic. Inherent to elaboration process, they are distributed inside the materials. These inclusions have generally a higher melting point than the host metals. Such inclusions in alloys reduce mechanical properties, are detrimental to surface finish and increase porosity, as well as having a tendency to increase corrosion. Further more, they act as stress raisers and can cause premature failure of in-service components.
There are many established methods of traditional NDE that are employed today to analyse the quality of new and in-service materials. The detection of inclusions is their prior task. From all of these, one can retain ultrasound, X-ray, eddy current or electrostatic conductivity [1]. Each of them has advantages and disadvantages. Successful results have been obtained using the previous mentioned techniques for non-metallic inclusions. However, metallic inclusions are a little bit more complex to detect due to the very similar physical properties they could have with the host metal.
In this work, we consider an alternative detection technique, which potentially has the possibility to detect surface and subsurface metallic inclusions, i.e. the Hot Tip ThermoElectric Power (HTTEP) measurement. The idea is to generate a thermoelectric circuit between the studied material and a tip which is temperature regulated [2]. A temperature gradient is induced and localised just under the tip. Due to the thermoelectric effect, a voltage is generated between the material and the tip. The knowledge of this voltage as well as the temperature gradient gives access to the local thermoelectric power. If a metallic inclusion is localised in the thermal gradient, the deduced TEP is modified, and then the inclusion is detected. In order to test the viability of this method, we also use numerical simulations that we have developed. We discuss about the potentialities as well as the possibilities of the HTTEP measurement.

2. Experimental and simulation details

2.1 The Hot Tip ThermoElectric Power device

Initially, this device was developed in order to measure the thermoelectric power (TEP or seebeck coefficient) of bulk materials. The principle is the following. Based on the seebeck effect, when two junctions made of two different metals have a temperature difference, an electromotive force is generated between the two junctions [2,3]. This voltage divided by the temperature difference is equal to the difference between the thermoelectric powers of the two metals. The knowledge of one of the two thermoelectric power leads to the determination of the second one. Basically, for the HTTEP device, one junction is realized by a small piece of copper which is not temperature regulated (see fig 1). Its role is to determine the temperature T_f and the electrical potential ϕ_f of the material to be measured [4,5]. It is referred as the cold touch. The second one has a truncated cone shape and is also made of copper. It is temperature regulated (T_m) using a heating element and is referred as the hot tip.

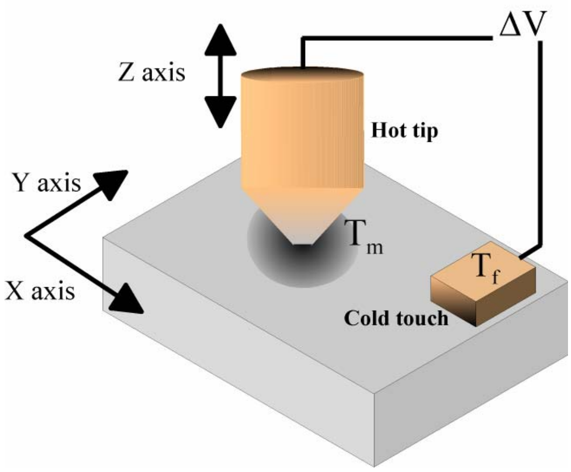

Figure 1: Schematic diagram of the hot tip thermoelectric power device

A detailed description of the device can be find in [5,6]. The contact surface is a circle with a diameter that can be change, for example, by machining the tip. For practical reasons, this value is generally ranging between 150 µm and 2000 µm. For our experiments, we use a tip with a diameter of 800 µm. By using a motor in the z direction and a force sensor, the contact between the material and the hot tip is perfectly controlled. With the help of a XY table, measurements of the TEP can be made at different positions, and then, maps can be plotted. For each measurement, thermal equilibrium must be reach and consequently, each point takes about 15 seconds to be acquired. For a good contact between the tip and the material, the precision of the device is less than 0.02 µV/°C.

2.2 Numerical model

In this paper, we briefly recall the numerical procedure we used. A detailed description of the model can be found in references [7,8]. We extend the procedure we used to the HTTEP measurements. Due to the symmetry of the problem (axisymmetric), calculations are only done in half of the plane (fig 2). The goal is to calculate the TEP of a structure made up of two metal phases (matrix and inclusions) whose thermoelectric distribution and properties are known. To determine the TEP, it is thus necessary to determine the temperature and the potential distribution within the material. The transport equations which govern the thermoelectric properties are coupled equations of heat and electricity in metallic materials.

In practice, to determine the temperature and the potential, it is enough to solve transport equations [7].

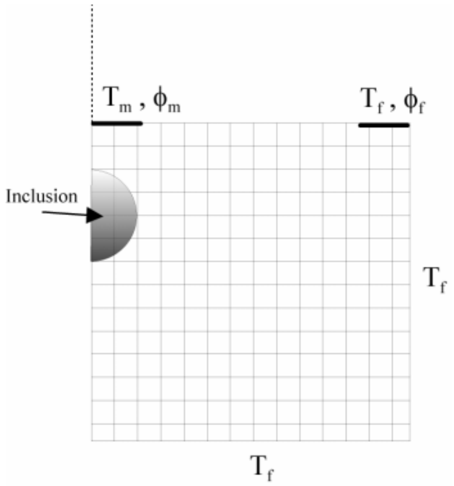

Figure 2: Discretization of the space. Hot tip contact and cold touch are modelled by a constant temperature.

We discretize 2D space by building a grid with a square mesh of dimension 300x300. Each mesh is represented by its co-ordinates and delimits a surface in which the temperature and potential are constant. The thermoelectric properties of each mesh depend on the material considered at the point. The temperatures of the hot and cold contact are kept constants, respectively at T_m=50 °C and T_f=20 °C. The external boundary is also kept at room temperature, i.e. 20°C, whereas Neumann conditions are used for the internal boundary.
First, we determine the temperature distribution within the sample using the conservation of thermal energy in each cell. By using the iterative method of Gauss-Seidel, we calculate the temperature distribution inside the material. We use a similar approach to determine the potential distribution within the structure in order to evaluate the thermoelectric power. Neglecting the thermal contact resistance, the apparent TEP results from the following relation:

$$TEP = \frac{\phi_m - \phi_f}{T_f - T_m} \qquad (1)$$

where ϕ_f and ϕ_m, are respectively the average potentials of the cold and hot contact, T_f and T_m the average temperatures of the cold and hot junction. As an illustration, the temperature distribution is plotted on fig 3 for a copper inclusion in an iron matrix. Most of the thermal gradient is located underneath the contact surface. The inclusion strongly modifies the temperature distribution, and thus makes the detection possible.

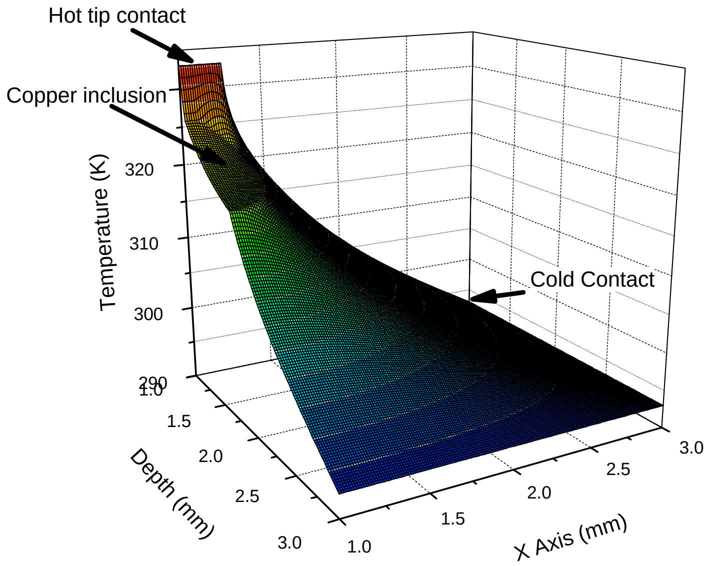

Figure 3: Temperature distribution under the hot tip contact.

2.3 Samples

In order to test the possibilities of the technique, we use two kinds of samples. The first one is composed of surface heterogeneities. Due to the segregation of alloying elements that can occur during solidification, some steels are composed of regions with a higher content of alloying element. Known under the name of ghost lines, these regions have different mechanicals properties in comparison with the matrix ones [9]. Detecting these heterogeneities is thus a prior task.

The second one are composed of artificial metallic inclusions in a copper matrix. For practical reasons, we choose a lead/tin metallic alloys for the inclusions. In a copper cube (50x50x50 mm^3), we have realized holes of 800 µm diameter using a drilling machine at different depths from the surface. The copper cube is heated in a furnace at a temperature of 450°C. Then, the holes are filled by simply melting the lead/tin alloys. Cooling down to room temperature and a final polishing ends the realization of the artificial inclusions. The physical properties of copper and tin/lead alloy are given in table 1.

Table 1. Thermoelectric properties of the two components

	Lead/Tin alloy	Copper
TEP (/ Copper tip)	- 2.9 µV/°C	- 0.3 µV/°C
σ	$5.85\ 10^6$ A/Vm	$0.567\ 10^6$ A/Vm
λ	51 VA/m °C	376 VA/m °C

3. Results and discussions

3.1 Surfaces heterogeneities

We have used the HTTEP device to detect heterogeneities located at the surface of the material. Since the thermoelectric power is very sensitive to the chemical composition [10], the HTTEP measurement has the potentiality to detect segregated areas. A map of the TEP values obtained using the HTTEP device is plotted on figure 4. For comparison, an optical micrograph is shown, after chemical etching with a Nital solution.

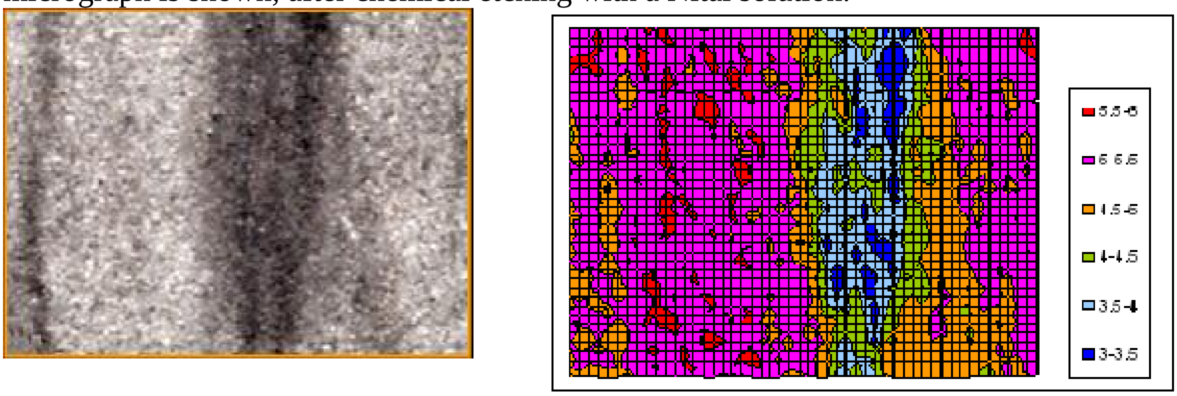

Figure 4 : Micrograph of ghost lines in a ferritic steel (left). HTTEP map of the corresponding zone (4mmx5mm).

A good correlation between the positions of the ghost lines and the lower values of the TEP can be observed. Segregated areas have lower thermoelectric power values due to a higher local content in allowing elements [10].

3.2 Subsurfaces inclusions

On figure 5, we have represented two lines profile of the measured TEP obtained with the sample containing a hole of diameter 800 µm and a centre located at 430 µm underneath the surface. It corresponds to a depth of 30 µm. The x axis represents the position of the hot tip. For each position, three points have been measured and between each point the tip have been successively lifted up and lifted down. The errors bars represent the maximum difference between the three values.

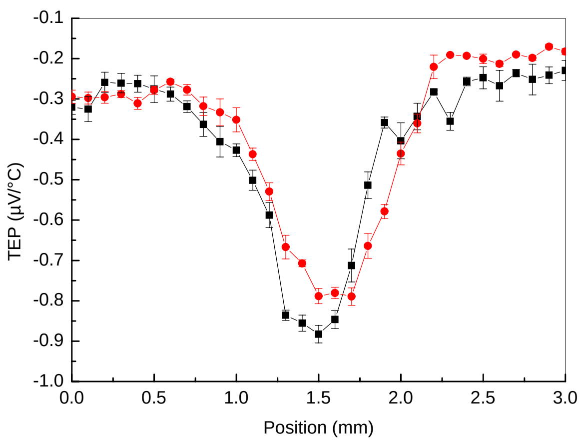

Figure 5: TEP profile near a subsurface inclusion. The inclusion is located at a position of 1.5 mm (depth 30µm). The two curves correspond to two profiles measured at a distance of 0.2 mm in the y direction.

The two scans correspond to a shift of 0.2 mm in the y direction. The artificial inclusions is located at 1.5 mm from the origin. A decrease of the measured TEP is observed closed to the position of the inclusion. The inclusion, due to a different value of its TEP modifies the measured TEP. Consequently, even if not visible from the surface, its presence is pointed out by the HTTEP measurement. To confirm this, we have made similar measurements for different depths of the inclusion. Results are plotted on figure 6. The difference in TEP between the copper matrix and the minimum value is represented as a function of the inclusion depth. Obviously, as the depth increases, the measured TEP is less influences by the inclusions, and consequently the difference decreases.
Numerical simulations have been performed in order to calculate the TEP for different inclusion's depth. The thermoelectric, electric and thermal properties used in the calculation are given in table 1. Results of the numerical calculations are compared with measurements values on figure 6. A good agreement is found. It confirms the role plays by the inclusion and its influence on the TEP, even for high depth.

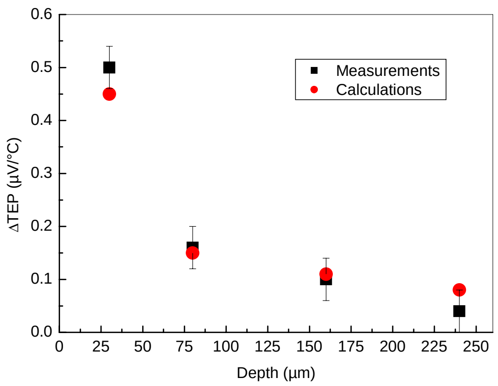

Figure 6: Measurements and simulations TEP results for different depth of a lead/tin inclusion in a copper matrix.

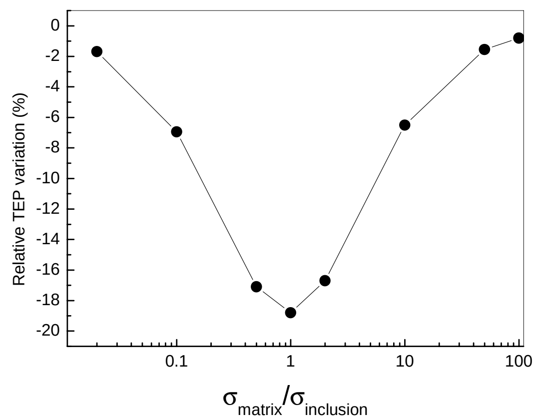

Figure 7: Effect of the electrical conductivity ratio on the relative TEP variation.

To check the size, the depth and the nature of inclusions that can be detect, we have performed complementary calculations. First, each inclusion is characterized by three parameters: thermal conductivity, electrical conductivity and thermoelectric power. For metallic inclusions, the two first ones are linked via the Wiedmann-Franz law [8]. Roughly speaking, it means that their influence on the TEP will be similar. For the thermoelectric power, the situation is very different, since it is totally independent of the two previous one. Basically, two metals with similar electrical conductivity can have very different thermoelectric power [2]. Hence, similar to what we have already shown in the case of composite materials [7], the two parameters that are going to be important are the electrical conductivity ratio and the difference between the thermoelectric power of the matrix and the inclusion. Obviously, it will be easier to detect an inclusion if its thermoelectric power is very different from the matrix one. The role of the electrical conductivity ratio on the TEP is less trivial. To quantify this effect, we have calculated for different electric conductivity ratio the relative variation of the TEP that can be measured for an inclusion with a centre located at 480 µm under the surface (depth 80 µm). Results of the calculations are shown on figure 7 for a ratio varying from 0.02 to 100.
In an interesting way, the maximum variation is observed when the electrical conductivity of the two metals are equal. Hence, an inclusion with an electrical conductivity equal to the matrix one can be easily detected as long as their thermoelectric powers are different.

In order to quantify the size and the depth of inclusion that can be detected, we have performed further calculations. For an electrical conductivity ratio equal to one, we have plotted on figure 8 the relative TEP variation (in %) that can be measured as a function of the depth and size of the inclusion. These calculations shows that bigger inclusions can be detected deeper.

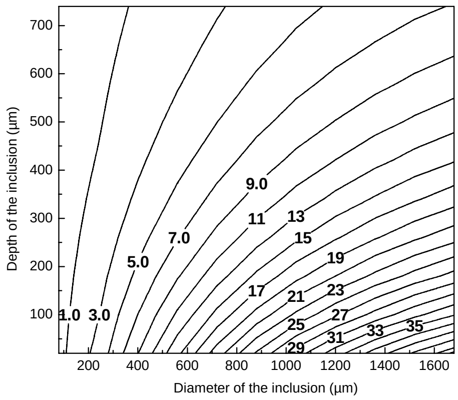

Figure 8: relative TEP variation for different diameters and depths of inclusion.

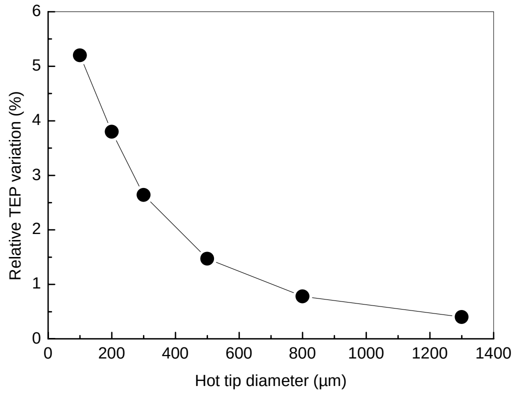

Figure 9: relative TEP variation as a function of the hot tip diameter.

Furthermore, if the TEP of the matrix and the inclusions are very different, small inclusions can also be detected. For example, for a sensitivity of the device of 0.02 µV/°C, and a difference in the TEP of 3 µV/°C, the relative variation that can be detected is less than 1 %. It means that inclusions of diameter 150 µm can be detected at a depth of 350 µm with a tip diameter of 800 µm (see figure 8). Detecting lower size inclusions can be perform in a simple way by reducing the diameter of the hot tip. This is confirmed by numerical simulations results plotted on figure 9. For an inclusion of diameter 100 µm located 50 µm underneath the surface, we have represented the evolution of the relative TEP variation as a function of the hot tip diameter. Such variation becomes more important and the detection easier as the hot tip diameter is reduced. This result confirms the possibility of probing different size of inclusions by changing the hot tip diameter.

4. Conclusions

From all the experimental and numerical results, we can say that the HTTEP technique can be successfully used for heterogeneities and inclusions detection. Not only surface particles can be detected but also subsurface one. The main advantages of this technique are as follows:

1. the possibilities of detecting metallic inclusion (heterogeneity) in a metallic matrix with a sensitivity all the more significant that their electrical conductivity are similar. Only the difference in the thermoelectric properties is important.
2. a high sensitivity for inclusions located closed to the surface.
3. the possibility of probing different inclusions size by using the appropriate hot tip diameter.

The main limitations are:

1. need to reach the thermal equilibrium. Each point takes about 15 seconds to be acquired. Works in the transient state are in hand in order to reduce this too long time.

2. being a contact measurement, the surface need to be relatively clean and smooth. Sample preparation is expected.

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