Int. J. Therm. Sci. (2000) 39, 519–525

S1290-0729(00)00231-3/FLA

Temperature profile measurements of near-field optical microscopy fiber tips by means of sub-micronic thermocouple [1]

Laurent Thiery [a]*, Nathalie Marini [a], Jean-Pierre Prenel [a], Michel Spajer [b], Claudine Bainier [b], Daniel Courjon [b]

[a] *Institut de Génie Energétique, Parc Technologique, 2 avenue Jean Moulin, 90 000 Belfort, France*

[b] *Laboratoire d'Optique P.M. Duffieux, Faculté des Sciences, 16 route de Gray, 25 030 Besançon cedex, France*

(Received 29 July 1999, accepted 30 September 1999)

Abstract — The present paper deals with the experimental measurement of temperature along various shapes of fiber tips used as near-field optical microscope probes. An ultra-small thermoelectric couple makes it possible to detect the tip temperature as far as the junction dimension remains smaller than the local investigated area. The couple is a 0.5 μm Pt and Pt–Rh 10% welded wires, whose dynamic and dimensional characteristics allow the investigation of the thermal behaviors of tapers under steady-state and periodic excitation modes (bandwidth near 1 kHz). Temperature profiles along these fiber tips (up to 400 μm from the apex) are shown and discussed. Confirming some authors' results, dramatic temperature increases due to an internal heating can occur for some configurations. Such temperatures can play a key role in the imaging process, because of the resulting behaviors of the apex, mainly due to its thermal expansion. Indeed, variation of the aperture diameter and thermoelastic interaction with the sample surface could explain some artefacts in the resulting images. © 2000 Éditions scientifiques et médicales Elsevier SAS

temperature / micro-thermocouple / optical fiber / microscopy / near-field / fiber tips

Nomenclature

a	thermal diffusivity	$m^2 \cdot s^{-1}$
e	aluminum layer thickness	nm
f_c	cut-off frequency	Hz
R_0	aperture radius of the fiber tip	nm
x	longitudinal coordinate along the fiber axis	μm
τ	time constant	s

1. INTRODUCTION

Scanning Near-Field Optical Microscopy (SNOM) which was born 15 years ago has encountered a growing interest because of its sub-wavelength spatial resolution [1, 2] (see Appendix). Numerous applications have been developed, such as local spectroscopy, fluorescence microscopy, optical storage (magneto-optics, nano-lithography), photochemical processes. The principle consists of scanning the sample surface with a small probe at a very short distance from the sample. The latter is kept constant by detecting the shear force exerted between the tip and the sample. Experimental and theoretical studies have demonstrated the dependence of the resolution on both the probe size (typically 10–100 nm) and the probe-to-object distance (typically a few nanometers). Various configurations have been used depending on the illumination mode and on the detection process:

- global sample illumination (in transmission, reflection, or total internal reflection) and local detection;
- local illumination;
- combined local illumination and detection by the same probe (reflection SNOM).

Various kinds of probes are available:

- micro-pipettes;

* Correspondence and reprints.
thiery@ige.univ-fcomte.fr

[1] Based on a paper presented as a plenary talk at Eurotherm Seminar No. 57 "Microscale Heat Transfer", Poitiers, France, July 8–10, 1998.

- tips on micro-cantilever;
- metallic wires;
- tapered optical fibers.

The last one can be used as a dielectric nano-antenna (apertureless detection) or can be metal-coated to create the nano-source which is necessary for some applications such as spectroscopy and lithography [3]. In this aim, a thin aluminum layer is deposited on the tapered fiber except on the very tip. The main consequence is a dramatic increase of the tip temperature, due to the light confinement inside the taper. This temperature level affects the probe-to-sample interaction process far before the risk of an irreversible damage. Besides, this thermal behavior prohibits the use of light power higher than 15 mW in the fiber. The analysis of the tip behavior for high temperature rates have been achieved some years ago [4, 5], but it is interesting to go on in more realistic situations where the harmonic temperature oscillations imposed by the light modulation (synchronous detection) have been taken into account. Moreover, only a very few experimental measurements [6] have confirmed the results on simulated thermal fields [7]. Finally, only steady-state thermal behaviors of the tip have been explored whereas most of the near-field techniques operate under periodic rates. The present paper deals with new experimental results obtained under both steady-state and periodic excitation rates. Temperature profiles along various fiber tip walls have been measured by means of a 0.5 μm thermoelectric couple, and the influence of parameters such as tip geometry or the modulation frequency is shown.

2. EXPERIMENTAL DEVICE

2.1. Fiber tip characteristics

A SNOM fiber tip can be obtained either by pulling and heating a single-mode fiber or by chemical etching (or both). The dimension and the shape of the fiber apex are highly dependent on the process and, more specially, on the duration of chemical etching. The first figure gives an example of a fiber tip obtained by such methods (*figure 1*).

For SNOM applications, such probes must be as thin as possible since the resolution is directly related to this parameter; the apex radius is generally between 10 and 100 nm. The whole taper is then coated with a thin aluminum layer (typically 100 nm), except the apex where an aperture of sub-wavelength dimension is formed by a shadowing effect created during the vapor deposition process. The interest of this metal layer is to prevent light leakage along the cone so that the light emission is reduced to a quasi-point source. Consequently, most of the input light is back-reflected, the transmitted part is very low (10^{-4}–10^{-7}), and the rest is dissipated into heat. Even for laser power in the range of 1mW, the resulting temperature is significant. Moreover, the quasi-Gaussian light profile combined with the conical shape of the tip induces a strong temperature gradient near the apex. Some authors have shown that even if the aluminum melting temperature is around 650 °C, the tip coating cannot bear more than 450 °C without damage [6]. The difference of the thermal expansion coefficients of the aluminum and the silica plays a basic role in the temperature limit, but also in the microscope behavior. Indeed, optical transmission variation and thermo-mechanical interaction with the surface may involve artefacts in the resulting images.

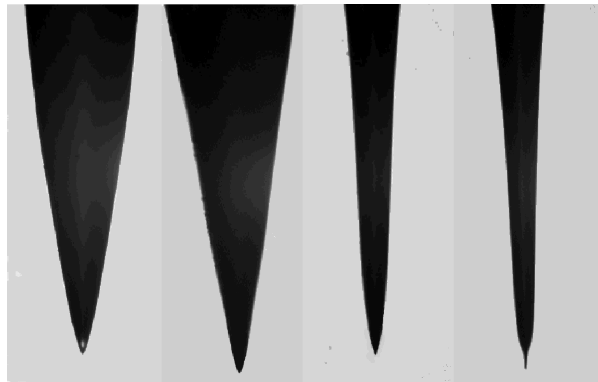

Figure 1. Some examples of possible fiber tip shapes (image width about 100 microns).

2.2. Temperature sensor

Temperature measurements are common on large samples when the heating is time independent. In order to increase the spatial resolution of the area under test, we have developed a thermoelectric sensor whose active region is small enough to fit the dimension of the fiber taper. For this we have realized welded wire thermoelectric sensors whose detecting diameter is as small as 0.5 μm. The wires are platinum and platinum-rhodium 10 % (S-type), they are welded by means of a sparking technique [8]. The main interest of such sensors is:

- only the junction will govern its behavior (unlike thin film deposition), therefore, the spatial resolution of the measurement is related to its volume (about 0.1 μm^3);

• the time response and the intrusive characteristics are related to its mass (about $2{\cdot}10^{-15}$ kg implying a heat capacity of several $pJ{\cdot}K^{-1}$). Although highly depending on the external conditions (convective and radiative thermal exchanges), the first order time constant of this kind of thermocouple is near 200 μs (about 800 Hz bandwidth).

About the use of such micron thermocouples we can mention photothermal microscopy, measurement of atmospheric temperature fluctuations, detection of acoustic temperatures, [9], etc.

As shown in *figure 2* the wires which are initially silver coated are then uncoated along several hundreds of micrometers. Before welding, these wires are introduced into a thin alumina ceramic tube, allowing the electric insulation and the protection of the junction (*figure 2*).

2.3. Experimental configuration

A laser beam, operating in the visible wavelength range at low power levels, is launched into the fiber core through a microscope objective (633 nm He–Ne or 514 nm Ar lasers). The light injection can be easily optimized by simply searching the maximum output power light. The usual positioners used in imagery are perfectly adapted to investigate the outermost millimeter of the fiber tip. In our case, the fiber is held by manual stages allowing various linear and rotating displacements. The temperature sensor is mounted on a three-axis motorized micropositioner including large and low travel ranges at low or high resolution (respectively DC motor and piezoelectric actuators). After setting the position of both sensor and fiber tip, the temperature measurement is performed by successive contacts of the thermoelectric junction with the taper along a generating line. As long as the junction remains much smaller than the local fiber size, one can consider that the measurement errors are only due to thermal contact resistance (typically a few percent). However, the temperature measurement at the very tip must be avoided for three main reasons:

- the cooling effect induced by the junction contact;
- the direct illumination of the junction;
- the fragility of the fiber apex.

As a consequence, measurements have been taken only 15 μm far from the apex where the cone diameter is more than a few micrometers.

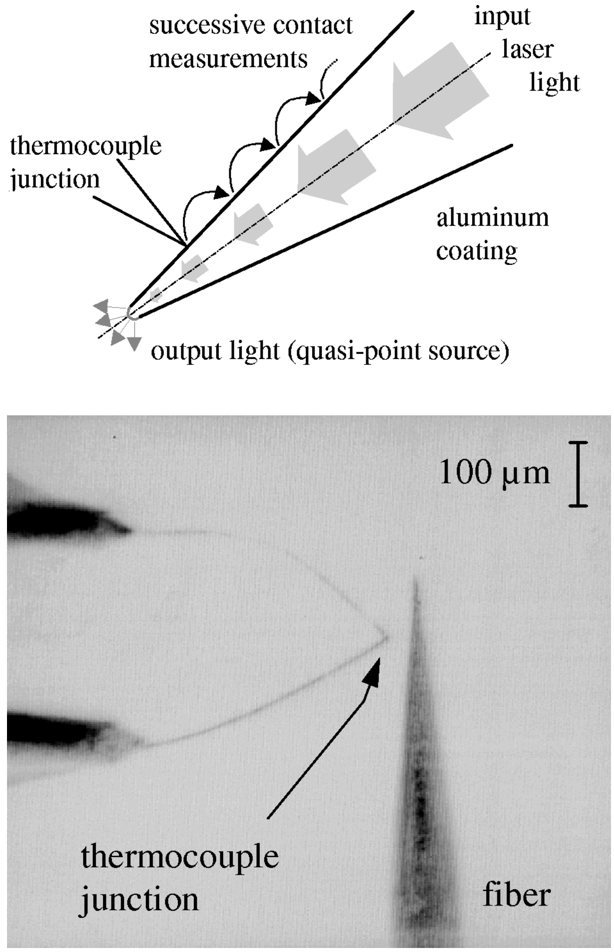

Figure 2. Experimental principle.

3. RESULTS

We present here the results obtained for three different shapes of tapers under various illumination conditions. The thermocouple has been connected to both a lock-in amplifier for extracting the oscillating component of the detected temperature and a low-pass filter amplifier (×1 000) for measuring the continuous component. Moreover, each point has been investigated under both steady-state and periodic excitation mode. Although the usual frequencies are of the order of 10 kHz in most of the imaging processes, the thermal inertia of both the sensor and the fiber itself has led to investigate the time behavior of the tip heating in a lower frequency range. It is clear that the thermal behavior of the tip is better described dynamically (i.e. in periodic excitation mode) than statically (i.e. in steady regime). *Figures 3–5* show a synthesis of these results. The periodic modulation is per-

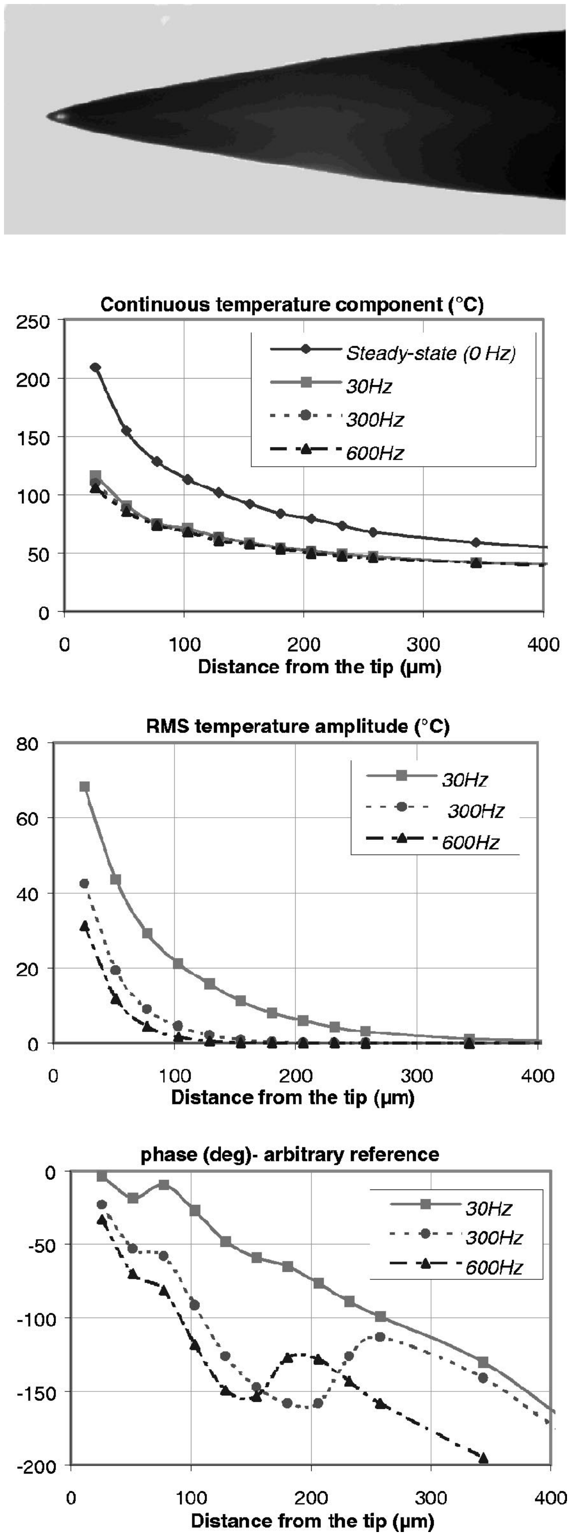

Figure 3. First fiber results obtained for 10 mW He-Ne (633 nm) input laser illumination.

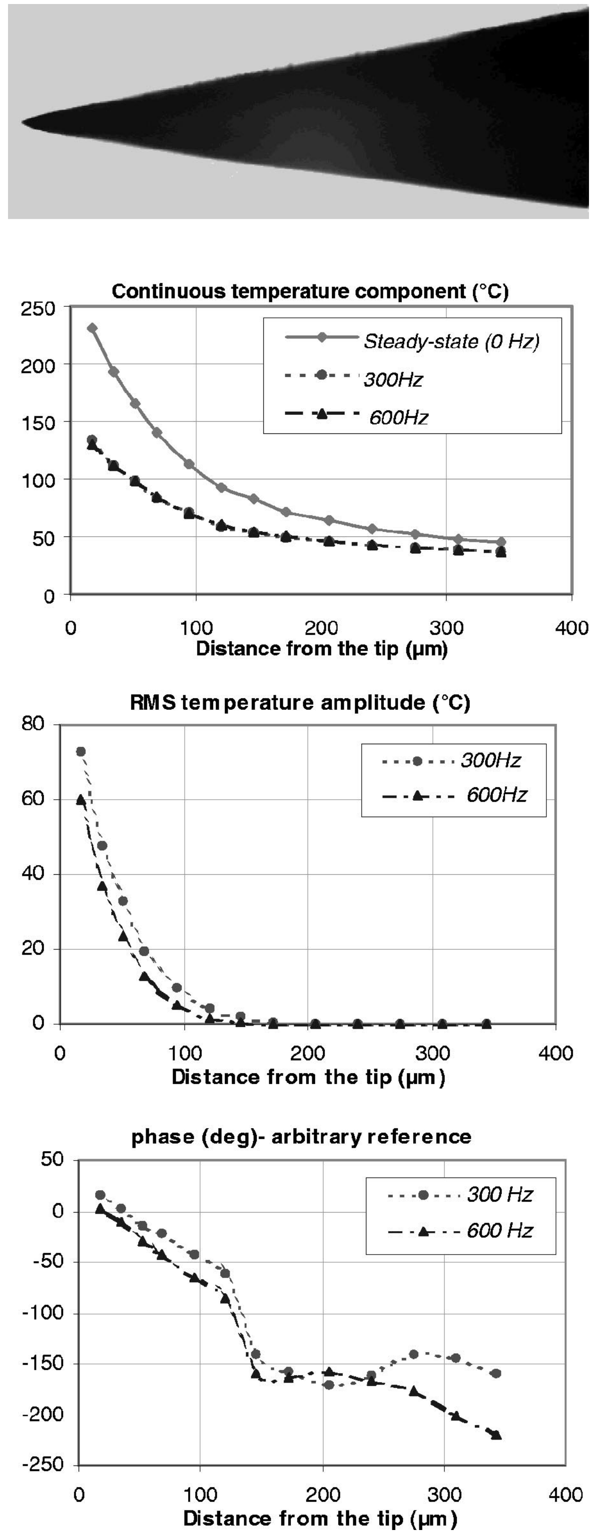

Figure 4. Second fiber results obtained for 6 mW Ar (514 nm) input laser illumination.

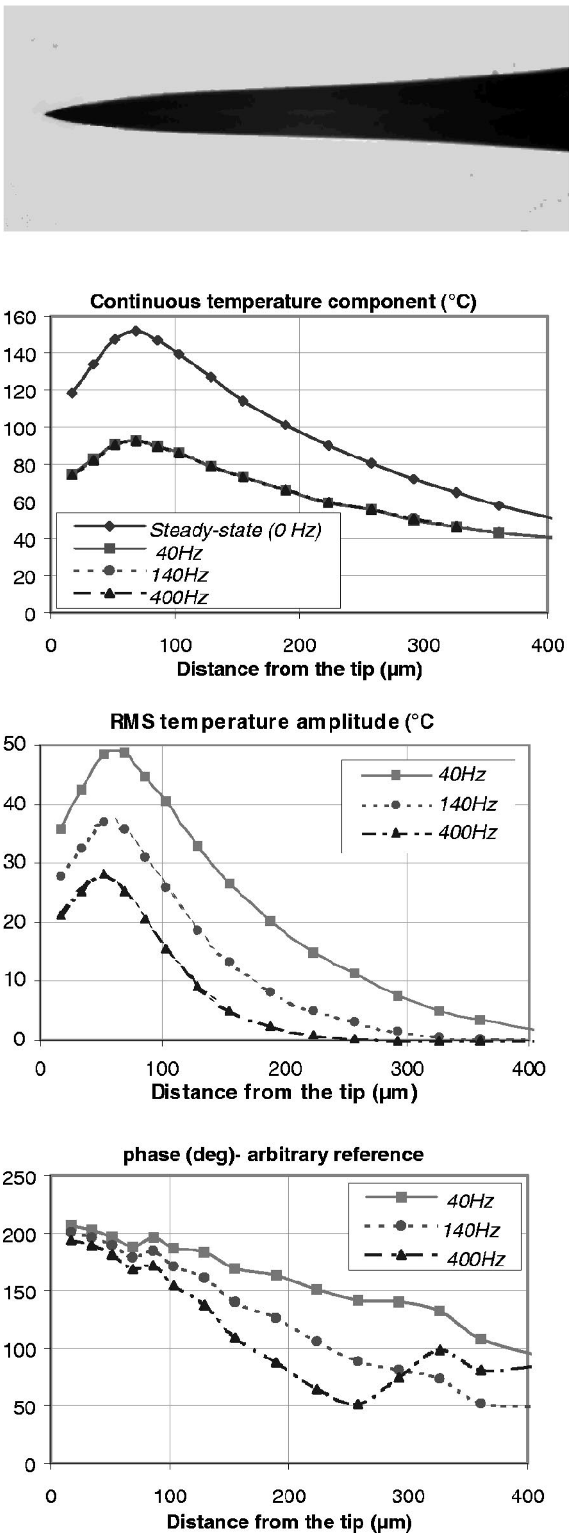

Figure 5. Third fiber results obtained for 6 mW Ar (514 nm) input laser illumination.

formed by means of an optical chopper. Let us note that because of lack of focusing of the Gaussian beam the effective injected light power was only about 40 % of the laser power.

Such results lead to the following comments:

- Continuous component amplitude is about half the steady-state regime one (0 Hz). It is merely a consequence of the chopper screening.
- The exponential-like decrease of the temperature along the fiber is in agreement with usual temperature measurements, especially for the harmonic components whose slope is stiffer and stiffer as the frequency increases.
- The temperature rate already observed [6] or expected [4, 5] by other authors is confirmed.
- Although we have not explored all the possible kinds of tapers yet, it is clear that the tip shape effect is preponderant in the thermal dissipation which is mainly due to a quasi-one-dimensional conduction process.
- The phase evolution of the harmonic components is rather complex. However, it is composed of several linear decreasing segments. Between these segments, some transition sections are observable whose origin may be due either to the topography or to a localized failure (no frequency dependence) or to changes in the heat transfer rate (frequency dependence). In the latter case, the thermal diffusivity is spatial dependent since the taper may be described as a composite medium whose thermal behavior is highly depending on the local fraction of aluminum coating width (*figure 6*). Indeed, according to Stahelin et al. [6], we consider that close to the very end, the thermal behavior of the tip is mainly governed by the aluminum coating, whereas for larger

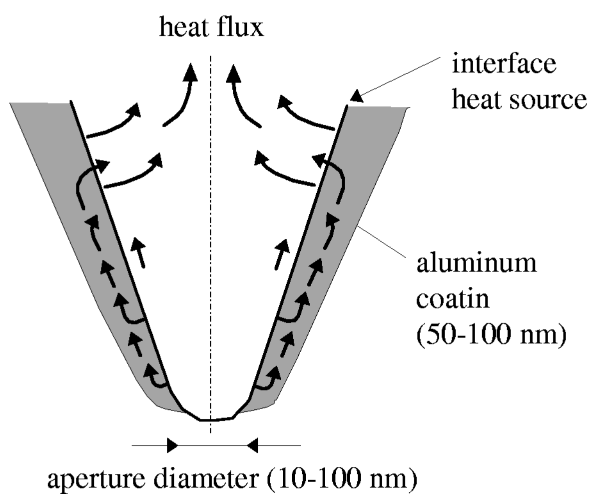

Figure 6. Schematic heat diffusion process in the tip end.

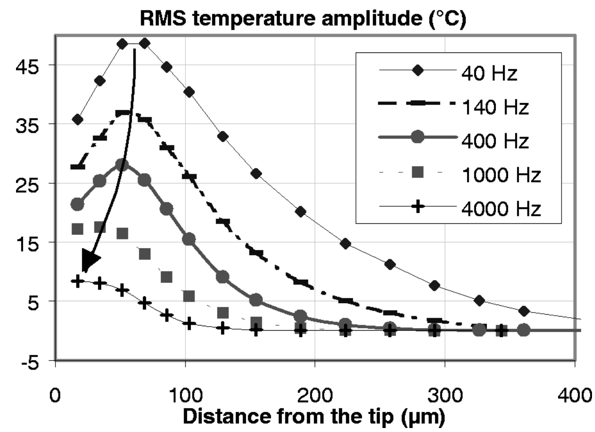

Figure 7. Harmonic amplitude of the third fiber temperature surface versus the frequency.

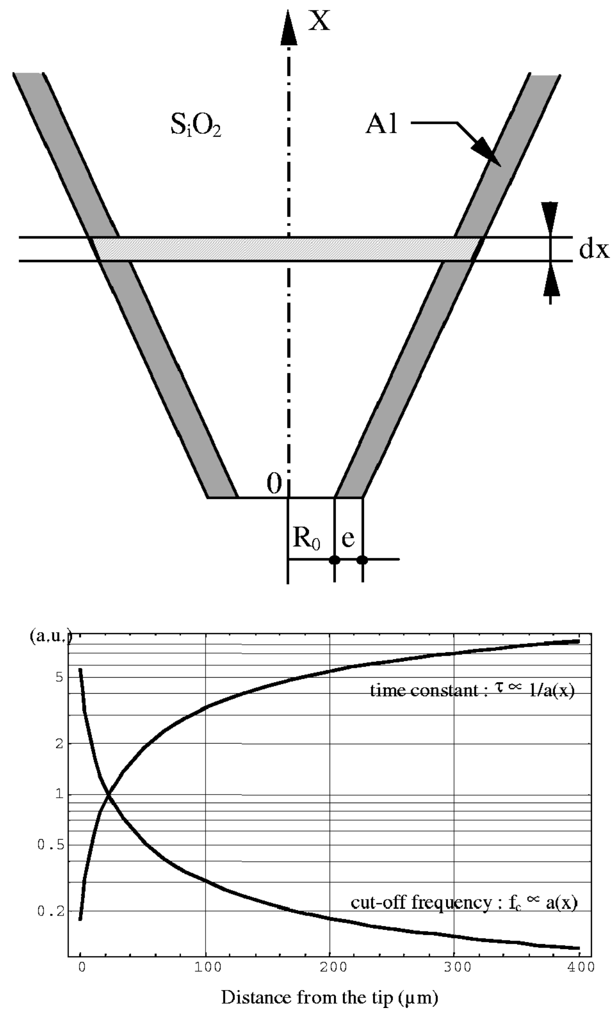

Figure 8. Simplified model of the taper and deduced dynamic behavior versus x.

distances the conductance of the SiO_2 core becomes more significant.

- Even if the temperature at the apex is not measurable, it is, however, possible to extrapolate for a rough estimate. Moreover, it must be noticed that the maximum temperature value is not systematically located at the apex, as shown in *figure 5* where the maximum continuous component value is obtained only 70 µm far from the tip end. This phenomenon, whose origin comes from both the injection light angle in the fiber and the tip shape involving a localized light concentration, has been theoretically predicted in some cases of illumination [5].

- The maximum amplitudes of the oscillating components are distance-shifted versus the frequency. This behavior has been confirmed by further measurements (*figure 7*) even if the sensor operated out of its bandwidth (limited to about 1 kHz).

To explain such a phenomenon, let us first consider that the temperature only depends on the location along the x-axis of the tip end. No radial temperature gradient could then occur. In our straightforward model, this part of the tip is assumed to be strictly conical. Then the heat balance equation is obtained by considering an elementary slice of width dx (*figure 8*). It is assumed that the thermal coupling of such an element with environment under harmonic excitation is mainly a conductive process inside the fiber core. The thermal behavior of any element is then related to its mass, which is all the more important that the considered element is far from the tip end ($x = 0$). Therefore, a thermal time constant (τ) or an equivalent bandwidth (corresponding to a first order thermal transient behavior) can be written as a function of the local thermal diffusivity $a(x)$ for each position along the tip. Consequently, the increase of the frequency will affect stronger the farthest part of the tip inducing a decrease of the temperature amplitude. The apex area requires higher frequencies to involve such a decrease of its local RMS temperature. The results of this phenomenon are shown in *figure 7* where a flattening of the curves is well visible. The decrease of the signal versus the frequency due to the sensor efficiency itself affects the whole curve globally.

- Finally, the results obtained for these three kinds of taper have shown the basic role of the cone shape in the temperature profile. Before definitely concluding on this point, we can predict some behaviors such as the maximum temperature value and its probable location.

4. CONCLUSIONS

The use of a sub-micron thermoelectric couple is an interesting way to measure highly localized temperatures and to extract transient components such as periodic ones in a bandwidth around 1 kHz. Applied to the crucial problem of tip heating in near-field microscopy our results are meaningful and complete the previous studies. Complementary measurements have to be performed in the framework of a wider synthesis. By considering a simplified one-dimensional heat balance of the taper as a composite medium ($S_i O_2$–Al), theoretical results have already been obtained, showing good agreement with experience. Indeed, even if the actual dimensions of such a taper can be amply sub-micronic, a simple macroscopic thermal modelling seems to be sufficient as a result of the large diffusion scale along the fiber core. These works will then be developed and analyzed in order to improve this first experimental approach, our aim being to provide a new knowledge on the role of tip-to-sample short distance interactions. Other characteristics should be soon investigated, such as mechanical consequences of these localized dramatic temperature increases. Finally, beside the problem of damage prevention, the mastering of the tip heating could lead to the emergence of new applications dealing with the handling of thermal mechanisms in the micro- and nano-world.

REFERENCES

[1] Pohl D.W., Scanning near-field optical microscopy, in: Sheppard C.J.R., Mulvey T. (Eds.), Advances in Optical and Electron Microscopy, Vol. 12, Academic, London, 1991.

[2] Courjon D., Bainier C., Near field microscopy and near field optics, Reports on Progress in Physics 57 (10) (1994) 989–1028.

[3] Davy S., Spajer M., Appl. Phys. Let. 69 (1996) 3306–3308.

[4] La Rosa A.H., Yakobson B.I., Hallen H.D., Appl. Phys. Lett. 67 (18) (1995) 2597–2599.

[5] Yakobson B.I., La Rosa A.H., Hallen H.D., Paesler M.A., Ultramicroscopy 61 (1995) 179–185.

[6] Stähelin M., Bopp M.A., Tarrach G., Meixner A.J., Zschokke-Gränacher I., Appl. Phys. Lett. 68 (19) (1996) 2603–2605.

[7] Kurpas V., Libenson M., Martsinovsky G., Ultramicroscopy 61 (1995) 187–190.

[8] Thiery L., Rev. Gén. Therm. 394 (1994) 551–561.

[9] Thiery L., Bailly Y., Lanzetta F., Gualous H., Gavignet E., Rev. Gén. Therm. 37 (1998) 60–73.

APPENDIX

Principle of scanning near-field microscopy (SNOM)

Scanning near-field optical microscopy is based on the exploitation of the peculiar properties of evanescent waves. The basic idea is the possibility of bypassing the diffraction limit imposed by the Abbe–Rayleigh criterium and, consequently, the possibility of getting sub-wavelength resolution. Because evanescent waves vanish exponentially over nanometer-sized distances, they have to be converted into propagating waves. In such microscopies the main element is the probe whose active part diameter is typically 50 nm. Its role consists in detecting the light beam emitted by an object a few nanometers from its surface, that is, in the near zone. Because of the short distance between the probe and the sample, the probe interacts strongly with the field emitted by the object. The resolution is mainly related to the probe size and the distance probe–sample. As any ordinary microscope, the near-field optical microscope can be used in transmission, reflection, internal reflection, in emission or collection mode. All these configurations have been explored for the last 15 years.

In our case, the probe is used for illuminating the sample and for collecting the optical signal containing the sub-wavelength information. It is basically a monomodal optical fiber which is tapered and metal coated except the apex. The so-created subwavelength aperture is used as a localized nano-source or/and as a local nano-detector.