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Application of DSPI to detect inhomogeneous heating on superconducting ceramics

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Abstract

This paper presents the first application of digital speckle pattern interferometry (DSPI) to detect inhomogeneous heat generation on a superconducting ceramic at cryogenic temperatures. The light scattered by the object is recorded with a CCD camera at the same time as a smooth reference beam. Comparison of two non-simultaneous frames provides information about the out-of-plane deformation field. Spatial phase shifting is used in order to get a good quality fringe pattern. The technique has been applied as a non-destructive evaluation of the performance of ceramic high temperature superconducting materials. DSPI allows the determination of the point where a hot spot will be generated with heating levels that do not deteriorate the sample properties. An excellent agreement between DSPI hot spot location and the position of the melting point that appeared in a destructive experiment has been obtained.

Keywords: digital speckle pattern interferometry, DSPI, inhomogeneous heating, superconducting ceramics

1. Introduction

One of the main technological problems of ceramic high temperature superconductors is inhomogeneous heating due to hot spot generation [1]. Heat dissipation starts at some points of the material where the superconducting properties are the poorest. Due to the low thermal conductivity of the material, power dissipated at these points produces local heating and it can deteriorate the sample properties, eventually reaching the melting point. In order to improve the development of technological applications with these materials, it is convenient to know the origin of these hot spots. Until now, the typical characterization of superconducting ceramics based on usual electrical transport measurements only gives information integrated along the length of the sample. The beginning of the quench can also be measured recording the bubble formation on samples immersed in liquid nitrogen [2]. Besides, in order to detect the hot spot generation, a high current passing through the material is usually required. A non-destructive technique that could detect where a hot spot will appear before excessive heat is generated would be greatly desirable.

In this sense, optical measurement techniques are becoming valuable tools due to their non-destructive nature. Digital speckle pattern interferometry (DSPI) is a well-established technique to measure small displacements of diffusely reflecting objects [3]. This technique produces fringe patterns by comparing two different specklegrams and has some interesting properties such as non-contact nature, digital recording, high sensitivity and full-field analysis.

In this paper, we report on the first application of DSPI to detect defects in a ceramic superconducting material during service, with the sample cooled to liquid nitrogen temperatures. Experimental difficulties, such as loss of coherence due to the nitrogen movement, that arise when working in these conditions have been analysed and solved.

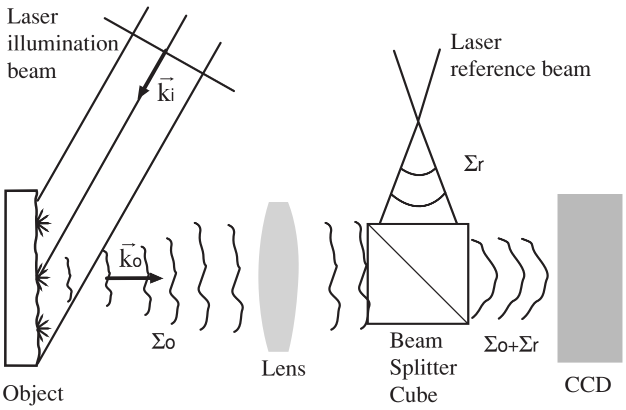


Figure 1. Basic optical arrangement for DSPI recording.

DSPI measures object deformations due to dilatation and/or displacements associated with the heating produced by the energy dissipated by the material. As the sensitivity of the technique is better than 1 μm, only a small temperature increase is required to produce a measurable deformation and the risk of damage in the sample disappears.

DSPI has been used to predict the location of hot spots in a superconducting ceramic with very low levels of applied current. Afterwards, in order to validate the method, the sample has been tested with high transport currents with the aim of producing enough heat to melt the sample. The melting has taken place at the position indicated by DSPI measurements.

2. Digital speckle pattern interferometry

In DSPI the interference between an object wave and a reference wave is recorded [3]. The interference pattern is known as a specklegram because of its speckled appearance. The light scattered by the object is the object wave and the reference beam is obtained by diverting a small amount of the main laser beam. Both beams are combined by means of a cube beam-splitter placed in front of the CCD sensor, as shown in figure 1. The specklegram is recorded at least twice, before and after having slightly deformed or displaced the solid object. The two specklegrams look indistinguishable since the object motion does not significantly affect the mean speckle size and intensity. However, significant changes in the intensity of each particular speckle can be observed. An image of the object surface overlaid by interference fringes is obtained by calculating the intensity difference at each pixel between these two specklegrams recorded for different states. The fringe pattern intensity is related to the local phase difference in the object wave, $\Delta\phi$. This phase difference can be expressed as $\Delta\phi = \mathbf{K} \cdot \mathbf{L}$, where $\mathbf{K} = (\mathbf{k}_o - \mathbf{k}_i)$ is the sensitivity vector, being $\mathbf{k}_i$ and $\mathbf{k}_o$ the local wave vectors of the illumination and observation beams and $\mathbf{L}$ the displacement vector. Thus, by measuring $\Delta\phi$, the deformation along the sensitivity direction can be inferred. However, the determination of $\Delta\phi$ from the intensity of the fringe pattern is only qualitative.

The introduction of phase shifting enhances the capabilities of DSPI. Phase shifting is a process in which known phase shifts are introduced between the two interfering beams, which allows the phase at each specklegram to be extracted from the value of the speckle intensity, without being affected by spatial non-uniformities in the beam intensities. The phase shift can be either a function of time or a function of position in the image. When the shift is a function of time, at least three interferograms must be recorded at discrete time intervals and the method is known as temporal phase shifting (TPS) [4]. A drawback of TPS is that random time-dependent phase fluctuations during the recording time of the phase-shifted frames caused by vibration, air turbulence or rapid object motion can spoil the measurement.

To overcome these problems, spatial phase shifting (SPS) can be used [5]. In this case, the phase of the reference wave is not shifted in time but in one spatial direction over the sensor. Only one specklegram is recorded and the intensity of three consecutive pixels is used as a set of equations. A conventional DSPI setup can be turned into a SPS-DSPI setup by shifting the origin of the smooth divergent reference wave with respect to the lens centre by a certain amount, Δx. This generates a linear phase shift in the x-direction of the sensor. As the phase-shifted data are recorded simultaneously on adjacent pixels, the speckle size must be increased up to about three pixels. In order for the sensor to resolve the modulation frequency, a maximum phase shift of 2π over three pixels has to occur.

The object phase map can be retrieved from each specklegram using a typical phase shifting formula based on the intensity value of three consecutive pixels. However, we prefer to use a global Fourier transform method (FTM) [6, 7], which better removes the unwanted noise. In this method, the first step consists in calculating the Fourier transform of the specklegram. Then, one of the two regions in the Fourier plane, whose centre is related to the carrier frequency introduced by the shifting, is selected. Finally, its inverse Fourier transform is calculated, thus obtaining the object phase and amplitude at each pixel.

The phase difference map is obtained by subtracting the object phase maps corresponding to two specklegrams. Let us remark that the phase difference is calculated with respect to some initial reference situation, which in general corresponds to the undeformed state. The calculated phase difference values lie in the range of 2π and values outside this range are wrapped. An unwrapping process, in which the phase is extended to a continuous range of more than 2π, is required for determining the deformation.

3. Optical arrangement

DSPI has been implemented for measuring ceramic superconducting materials during service as shown in figure 2. Due to the dynamic nature of the process only SPS can be used. The sample is illuminated at an angle of 45° by a collimated laser beam from a 17 mW He–Ne laser and imaged onto the sensor of a CCD camera by a lens of 120 mm focal length. The angle between the illumination and recording directions is 90° at the object surface. With this setup, the phase difference $\Delta\phi$ gives information mainly on the out-of-plane displacement; the sensitivity of the technique has been determined to be 0.45 μm per fringe. An aperture of $f/16$, a magnification of 0.28 and a standard 652 × 494 pixel camera are used for the recording. With these experimental conditions, the recorded

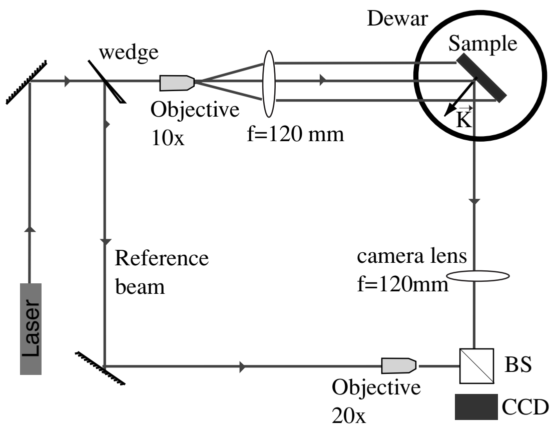


Figure 2. Schematic top-view of the experimental setup.

object area is 23.9 × 13 mm^2. Let us note that since the object surface is not parallel to the camera, the recorded object length is proportionally bigger in the horizontal direction than in the vertical direction.

The reference beam is divergent with the focus at the same distance from the sensor as the camera lens aperture. For achieving the spatial phase shifting, the origin of the reference wave is transversally displaced by about 3 mm in each direction. For this purpose the 20× microscope objective is placed on a micrometric movement stage.

Figure 3(*a*) shows a typical image where a length of 23.9 mm from a 40 × 7 mm^2 sample has been recorded. The recording magnification has been chosen so that the object covers a significant part of the CCD sensor height, but magnifications at least two or three times smaller could have been used. Figure 3(*b*) shows a typical specklegram for the same object and magnification. Let us note that the main features on the specklegram do not correspond to a typical speckle field; they are produced by non-uniformities in the reference beam. The non-uniformities are bad enough for preventing the standard DSPI from working. However, the SPS-DSPI is robust enough to produce good phase maps. Figure 3(*c*) shows the Fourier transform of the specklegram; the bright and wide central area is mainly due to the reference beam non-uniformities because the intensity of the speckle halo is much lower. The object phase and amplitude is to be obtained from either of the other two not so bright regions.

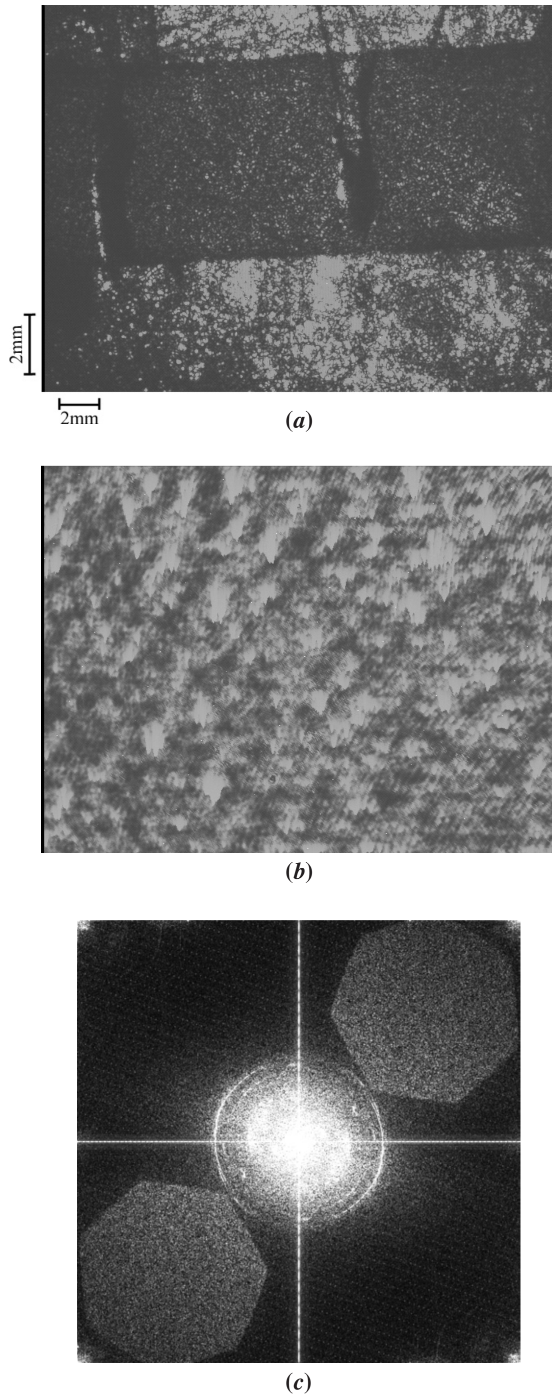
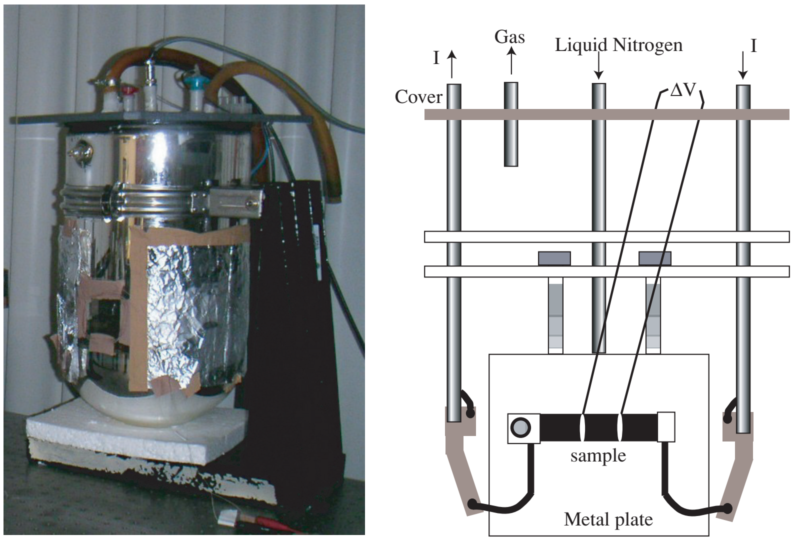


Figure 3. (*a*) Photograph of the sample, showing the field of view; (*b*) its corresponding specklegram; (*c*) Fourier transform of the specklegram.

4. Cooling arrangement

As the operating temperature for this kind of ceramic superconductors is below 90 K, the sample can be cooled down with liquid nitrogen. A glass dewar (figure 4(*a*)) with a height of 420 mm and a diameter of 200 mm has been designed for this purpose. An 85 mm high section located at 200 mm from the top and all along the dewar perimeter has been left clear to allow wide optical access for several optical techniques. In the present experiment, optical access is only needed for the illumination and observation of the sample. Thus, only two windows at 90° have been left by covering the rest of the uncoated section with aluminium foil to reduce the ambient heat radiation entering the dewar. Each window has been heated with an external manganin resistance to avoid condensation on the external wall. Condensation not only reduces the optical transmittance but even prevents DSPI from working.

The sample is attached to an aluminium plate held from the top cover of the dewar (figure 4(*b*)). Two silver current contacts have been placed on the sample in order to apply a transport current, *I*, higher than the critical current value, I_C, where dissipation starts. The electrical wires come from the top but are immersed in the nitrogen before contacting the



(*a*) (*b*)

Figure 4. (*a*) Image of the glass dewar where experiments have been performed. (*b*) Schematic diagram of the sample holding arrangement.

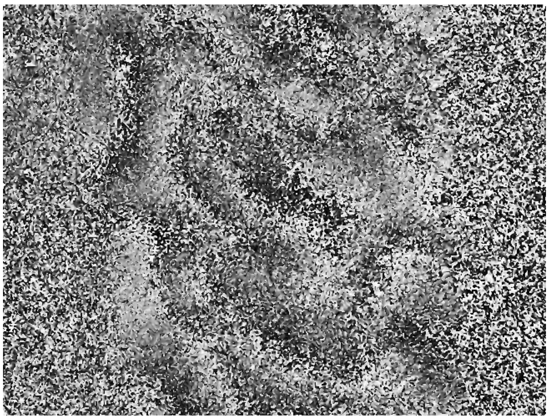


Figure 5. Phase difference map obtained from an undeformed object with the object beam travelling 20 mm in liquid nitrogen.

superconductor to minimize the amount of heat transferred. Another couple of thinner wires are attached to the sample for measuring the voltage drop between them, ΔV. This four-probe arrangement allows *in situ* electrical characterization of the material (i.e. resistance and critical current). The left end of the sample is fixed by a screw to avoid bulk movements.

The traditional way to cool the superconductor to temperatures below 90 K is by direct immersion in liquid nitrogen. Since the sample is located at the centre of the dewar, the object beam travels about 200 mm in liquid nitrogen, close to its boiling point. In these conditions, temporally and spatially random phase variations in the object beam are produced due to the non-steady state of the liquid nitrogen. These random phase variations should decrease both when decreasing the path length in nitrogen and when reducing the liquid nitrogen movement. We have checked that, for a path length of 200 mm and even when vacuum is made on the dewar so that nitrogen in a more steady undercooled state is present, the phase variations are big enough for the phase maps from consecutive specklegrams to be totally uncorrelated. Figure 5 shows the phase difference map obtained when decreasing the path length in nitrogen down to 20 mm. A 4 mm wide metallic part of the sample holder has been used as the object. Several specklegrams at 400 ms intervals with 2 ms exposure times were recorded with this object, always in its undeformed state. Complex and continuously increasing noisy phase difference maps were systematically obtained. After a few seconds, the noise was too much for any pattern to be seen in the phase maps.

As the sample must be located at the centre, it is not feasible to have a short enough path length in liquid nitrogen. Since phase variations are much smaller in gaseous media than in liquid media, the liquid nitrogen should be removed from the object beam path. In that case, the sample should be cooled down by conduction. This is the option we have chosen for the present experiments. The sample has been thermally fixed to a big aluminium plate ($100 \times 120 \times 5$ mm^3) with its lower part immersed in liquid nitrogen, whose level is kept just below the sample. A piece of paper is placed between the sample and the plate in order to maintain electrical insulation. The thermal contact has been improved by using a layer of APIEZON® N grease. In this case, the near boiling liquid nitrogen still introduces an unwanted fringe pattern (figure 6(*a*)). Although this pattern is less complicated than before, it is enough to hide any phase change due to the sample deformation. Furthermore, although the phase maps get noisy (i.e. the specklegrams get uncorrelated) slower than before, the correlation is maintained for only a few minutes. However, after making a rotary pump vacuum ($\sim 10^{-1}$ atm) inside the dewar, very good phase difference maps (figure 6(*b*)), without any undesired pattern, are obtained for at least half an hour.

Let us note that, although some discontinuity in the phase difference map might be observed on the borders of the sample, it is difficult to precisely know the position of the sample in the map. This problem has been overcome by taking a sample photograph (like the one shown in figure 3(*a*)) in each experiment. The black lines in the phase difference maps

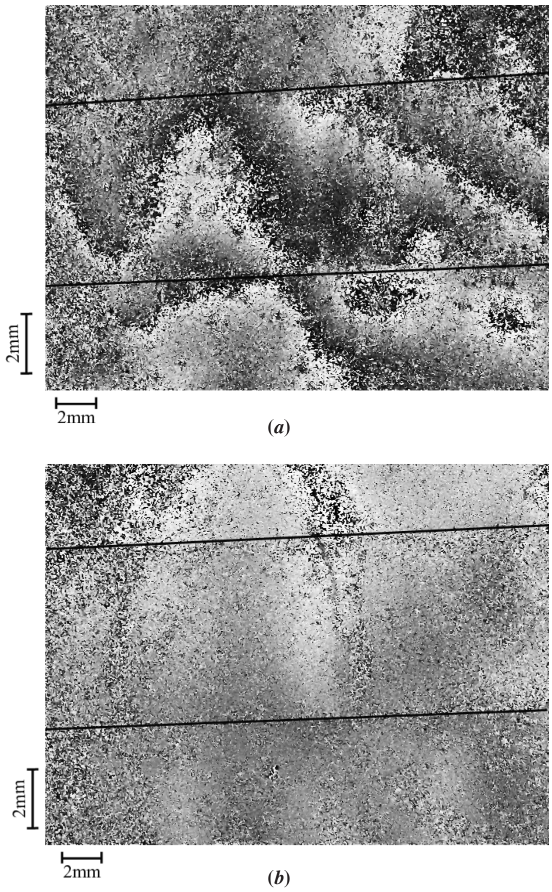


Figure 6. Phase difference map obtained when the undeformed object is in a nitrogen atmosphere at (*a*) atmospheric pressure and (*b*) low pressure.

(figures 6–8) show the sample limits as obtained from the corresponding photographs.

5. Experimental results

DSPI has been tested on Bi-2212 high temperature superconducting monoliths. The samples were textured with a laser floating zone (LFZ) technique [8] using a high power continuous diode laser emitting at 890 nm. Both surfaces of the sample have been textured in order to avoid the bending of the superconducting ceramic during the annealing [9] due to the differences in density between polycrystalline precursors and the textured material. The samples show some anisotropy in their texture and superconducting properties. The best textured regions can be found at both surfaces of the samples. The texture continuously deteriorates when approaching the sample centre. Close to this centre, a large number of defects, bubbles and non-superconducting phases are present. They have been originated during the laser processing.

To simplify the current setup and minimize the heat generated at the electrical contacts, DSPI has been initially tested in samples whose critical intensity at 77 K, I_C(77 K), is lower than 15 A. A series of specklegrams at 400 ms intervals with 2 ms exposure times was recorded.

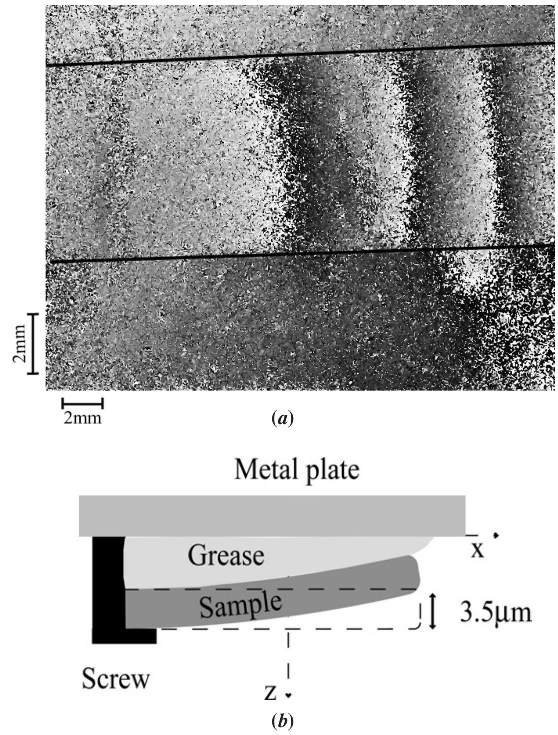


Figure 7. Results on uniform heating. (*a*) Phase difference map for $I = 2$ A. (*b*) Schematic of the corresponding sample deformation.

The first experiment was performed at room temperature, where the sample is in the normal state (non-superconducting state), with a resistance value of 28 mΩ. When a small current is introduced in the sample, uniform heating is expected. We introduced up to 2 A and obtained sets of parallel fringes (figure 7(*a*)). The main contribution to these fringe patterns comes from the material bending due to the temperature difference between the rear and the front surfaces. The rear surface, in contact with the metal plate, has a lower temperature than the front surface producing a bending as shown in figure 7(*b*). Other possible contributions coming from electromagnetic forces are negligible in this case due to the low value, slowly changing involved currents. The temporal evolution of the fringe pattern shows that the number of fringes increases with time until a final stationary state is reached. This is in agreement with the time evolution of the material resistance, which is proportional to the temperature variations on the sample. In these experiments, the temperature difference on the sample increased about 12 K at the end of the measurements. The maximum out-of-plane displacement in the field of view was 1.8 μm, which can be extrapolated to be about 3.5 μm in the sample free end. In these experiments, the sample perfectly recovers its undeformed state shortly after switching off the current.

The next experiments were done at temperatures between 77 K and 90 K, where the samples are in the superconducting state. When the applied current is below the critical value, I_C, the resistance is almost null and no heating can be observed. When the current is bigger than I_C, the point with the poorest superconducting properties starts to dissipate. Thus, a local temperature increase is produced and the adjacent points can

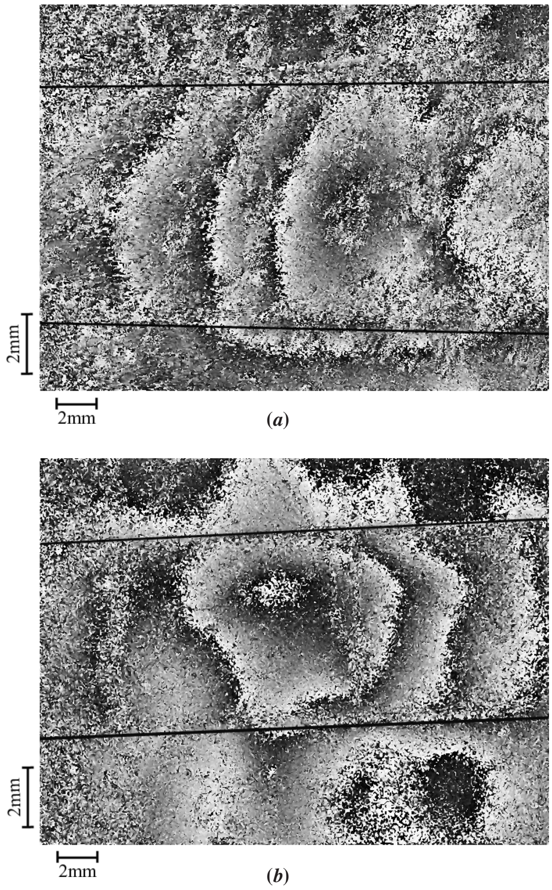


Figure 8. Phase difference maps obtained at low temperature with $I \approx 3I_C$ from (*a*) a sample with an induced defect and (*b*) a typical superconductor sample.

reach the nonsuperconducting state and start to dissipate too. The heat propagation rate is very low due to the very low thermal conductivity values of the material.

Figure 8(*a*) corresponds to a sample with a defect in the front surface. The defect was produced intentionally by removing the laser processed material along a transversal line. The critical current $I_C(77\text{ K})$ went down to 15 A, which is 30% of the original undamaged sample value. The phase difference map was obtained with $I = 10$ A at a temperature such that $I_C(T) = 2$ A. The circular pattern shows that the local heating has produced a 'bulge', the centre of this bulge being at the same position as the induced defect. Figure 8(*a*) also shows some small phase variation outside the ceramic probe, which could be produced by thermal or mechanical displacements of the supporting metal plate or by thermal variations of the surrounding nitrogen atmosphere. In any case, this phase variation is small enough (less than one fringe) so that it does not disturb the hot spot detection process. The sample could be tested many times without being destroyed because the local temperature changes needed to detect the defect were about 100 K, which do not deteriorate the ceramic. In fact, we have checked that all the fringes disappear from the phase difference maps sometime after switching off the applied current, which probes that both the ceramic probe and its supporting metal plate perfectly recover their initial states.

Figure 8(*b*) corresponds to a typical sample whose $I_C(77\text{ K})$ was about 10 A. As usual, I_C decreases when T increases. The superconducting properties of the material are determined by the quality of the intergranular junctions between the superconducting grains. As in any ceramic material, the sample presents a distribution of intergranular properties. Dissipation starts at the worst points of the material. The phase difference map was obtained with $I = 6$ A at a temperature such that $I_C(T) = 2$ A. Again, a 'hilly' deformation is apparent; its centre marks the position of the most important defect. As a posterior confirmation of the defect position, another experiment was performed with pulsed currents up to 100 A over 500 ms. In this case, a local melting took place at the same point indicated by DSPI. This demonstrates that DSPI, although only sensitive to displacements of the front surface, can also detect the local heating produced inside or even at the back of a sample 1.4 mm thick, which greatly increases the defect detection capabilities of this technique.

6. Conclusions

Digital speckle pattern interferometry (DSPI) has been demonstrated as a non-intrusive technique to detect the localization of inhomogeneous heating generation in high temperature superconducting ceramics. Experiments at room temperature and close to liquid nitrogen temperatures have been performed. In the first case, as uniform heating is produced, a set of parallel fringe patterns is obtained associated with bending of the sample. In the second case, the sample is in the superconducting state and the fringe pattern shows clearly the centre of a hot spot. In both cases, the produced heating levels are low enough for the sample not to deteriorate. As a consequence, DSPI can be used as a complementary non-destructive technique to study the origin of these hot spots in ceramic superconductors, that once located can be thoroughly investigated by other experimental techniques.

Acknowledgments

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