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Publisher's version / Version de l'éditeur:
https://doi.org/10.1016/j.expthermflusci.2005.05.004
Experimental Thermal and Fluid Science, 30, 2, pp. 153-159, 2005-11

A Fast-Response Thin-Film Thermocouple to Measure Rapid Surface Temperature Changes

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Abstract

A thin film thermocouple has been developed to measure rapid surface temperature change. A fine wire of Constantan was inserted vertically into a hole drilled through a steel plate and held in place by ceramic cement that acted as an electrical insulator. A thin conductive film was deposited on the surface to provide an electrical connection between the steel substrate and the thermoelectric wire. The voltage difference between this junction and a second junction kept in an ice bath was calibrated as a function of the surface temperature. Tests showed that the thin film sensors could detect a temperature rise of over 200°C in less than 10 ns produced by a laser pulse focused on the junction. The sensors were also used to measure transient surface temperature distribution under an impacting droplet of molten aluminium.

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1. Introduction

Engineering applications frequently require measuring temperature variation of surfaces exposed to a fast changing heat flux. Typical examples of surfaces experiencing rapid temperature fluctuations include engine cylinder walls; hot-rolled metal sheets quenched by water sprays; and dies used for casting metal parts.

Temperature measurement of a steel surface onto which a droplet of molten metal falls is a problem of particular interest to us. Such data have been used to estimate thermal contact resistance between liquid metal and a steel substrate [1,2]. The entire process of impact, spreading and solidification last a few milliseconds; to record temperature variations accurately requires a temperature sensor with a response time of only a few microseconds. Previous investigators [3] have used optical pyrometers to measure droplet temperature evolution, but the response time of such sensors is 1 ms or greater, so that temperatures measured are those after droplet solidification is complete.

Aziz and Chandra [1] used a commercially available thermocouple (E-123K, Nanmac Inc, Framingham, MA) that consisted of two thin ribbons of Chromel and Alumel separated by an insulating sheet of mica, enclosed in a stainless steel sheath. The thermocouple was inserted vertically through the test substrate and ground flush with the surface, forming a bare thermocouple junction where the thermocouple materials smeared across the insulating mica layer and touched each other. The thermocouple response time, as reported by the manufacturer, was 10 μs, making it suitable for measuring surface temperature variation under impacting tin droplets.

The Nanmac thermocouple, though useful in measuring surface temperature variation, has several limitations. The thermocouple sheath has to be made of the same material as the substrate, limiting the number of materials that can be used. Attempts to change surface roughness by grit blasting or polishing the surface destroy the thermocouple junction, so the effect of surface roughness on thermal contact resistance cannot be studied. Since the sheath is over 3 mm in diameter it is not possible to place sensors close enough to measure spatial temperature variations under an impacting droplet.

Kim et al [4] used electronic fabrication techniques to place resistance temperature sensors close together on a surface under an impacting water droplet. However, such sensors require that the substrate be an electrical insulator and they are too delicate to survive contact with molten metal.

We developed a thin film thermocouple to measure the temperature variation of a steel plate when molten aluminium droplets landed on it. Our objective was to develop a temperature sensor that: *a*) had response time less than 1 μs; *b*) was rugged enough to withstand contact with molten aluminium; *c)* could be built on both rough and smooth surfaces of any conductive material; and *d*) was simple to fabricate. This paper describes the construction of the thermocouple, and tests done to characterize its performance.

2. Thermocouple Fabrication

Figure 1 shows a schematic diagram illustrating the principle behind the thin film thermocouple. The conductive steel substrate acted as one of the thermocouple materials. A fine wire of a second thermocouple material (such as Constantan, copper or Chromel) was inserted through a hole drilled in the substrate and held in place by ceramic cement that acted as an electrical insulator. A thin conductive film was deposited on the surface to provide an electrical connection between the steel plate and the thermoelectric wire. The voltage difference between the two junctions formed where the conductive film contacted the steel substrate and the wire, and a third junction kept in an ice bath at 0°C (see figure 1) was a function of the temperature of the thin film.

Figure 2 shows a top view of three temperature sensors apart on a steel plate. In this particular case the test substrate was made of H-13 tool steel into which three 0.57 mm holes were drilled with centre-to centre spacing 1.15 mm. Constantan wires (0.25 mm diameter, SPCI-010, Omega Engineering Inc., Stamford, CT) were inserted into these holes, insulated from the surrounding metal by ceramic cement. A piece of graphite was drawn across the surface, leaving a thin conductive film that made an electrical connection between the wire and surrounding steel.

This thermocouple design offers several advantages. It gives a true measurement of the temperature of the surface where the conductive film is applied. The film can be very thin (typically 0.1 to 2 μm) to give very fast sensor response time. The size of the temperature sensor is small (approximately 0.5 mm diameter, see figure 2), giving a localized temperature measurement and allowing several sensors to be placed close together. The length of the sensing film is so small (<0.2 mm) that temperature variations across it are typically negligible.

Figure 3 shows a detailed view of the thermocouple assembly. Substrates used in our experiments were 50.4 mm square, 6.35 mm thick plates, made of either 303 stainless steel or H-13 tool steel. Several 0.57 mm diameter holes, one for each sensor to be built, were first drilled through the plate using a tungsten carbide drill bit. Only one hole is shown in figure 3 for clarity. Then a 9.3 mm diameter hole was drilled part way through the plate and tapped with a 1/8"-27 NPT taper thread allowing a pipe fitting (Swagelok SS-300-1-2BT) to be connected. All the small holes were located within the 9.3 mm diameter of the larger hole. A small amount of ceramic cement paste (OMEGA CC High Temperature cement) was forced into each of the 0.57 mm diameter holes from below. Lengths of 0.25 mm diameter wire were inserted into the bottoms of the holes and pulled through so that at least 25 mm protruded above the surface. To ensure that the wires were insulated from the surrounding metal the electrical resistance between them and the steel plate was measured. The cement was allowed to dry for at least 20 hours.

Once the cement had hardened the wires were threaded through a length of alumina tubing with multiple bores, one for each wire. The alumina tubing was passed through the pipe fitting and held in place with a Teflon ferrule. Sand paper (60 and 80 grit) was used to polish the steel plate and remove any excess cement until the surface was flat. Further sanding or grit blasting was used to produce the desired surface roughness.

To complete the thermocouple circuit it has to be connected to a reference junction and voltage measurement instrument as shown in figure 1. The steel plate was connected to the reference junction with a wire made of the same material. In the case of the H-13 tool steel plate, where we could not obtain such a wire, we machined off a long helical ribbon of H13 steel using a lathe and used that in place of a wire. The reference junction was maintained at 0°C, in an ice and water bath.

Several different wire materials were tested, producing a wide range of voltages. Table 1 lists the voltage produced by a thermocouple composed of a steel plate (either 303 stainless steel or H-13 tool steel) and wires of different materials. The hot junction was maintained at 200°C and the reference junction at 0°C in all cases and a graphite film was used to connect the plate and wire. The greatest output in all cases was with Constantan as the thermocouple wire, and this was selected for all further tests.

Different materials were also tested to form a film between the Constantan wire tip and steel plate. The simplest technique was to take a sharpened graphite rod and to lightly touch its tip on the sensor. This could be done in a repeatable manner by measuring the electrical resistance between the wire and surface and applying enough graphite to give a resistance of 30–70 Ω.

Metallic films can also be used to make the temperature sensor. We used silver and platinum inks (SPI Supplies, West Chester, PA) applied with a brush. A copper film was applied by placing a drop of copper sulphate solution on the junction and applying a 12 V DC electric potential between the substrate (kept as the cathode) and a copper wire immersed in the droplet. A thin copper film deposited on the surface.

All these techniques successfully produced a thin film temperature sensor. Calibration tests showed that the voltage produced was independent of the material used for the film. The length of the film was so small (< 0.2 mm) that its two ends were at the same temperature. The thermoelectric potentials generated at the end junctions therefore cancelled each other, so that the connecting film material had no measurable effect on the thermocouple output. The decision on which coating to use depends only on its durability. In our application we found that exposure to molten aluminium quickly destroys metallic films; graphite was much more durable and easy to apply and was used in all our experiments. Interestingly, the molten metal itself completed the thermocouple circuit and generated a voltage even when the thin film was destroyed. However, once the metal solidified electrical contact was lost and the temperature sensor no longer worked.

Film thickness was estimated by measuring the surface area of the film under a microscope and its electrical resistance. Knowing the specific resistivity of the material and assuming the film was uniform, its thickness could be estimated. Thicknesses varied from 0.1 μm to 2 μm in our test and this variation had no effect on the sensor output voltage.

3. Measurement and Calibration

Using the highest output thermoelectric materials (steel-Constantan, see table 1) the output from the thermocouple was of the order of tens of millivolts. Accurate measurement required either amplification of the signal or a data acquisition system sensitive enough to measure such low voltage levels. Also, careful attention has to be paid to how electrical connections are made since long thermocouple wires act as antennae that pick up noise and ambient interference such as electro-magnetic interference (EMI) and radio frequency interference (RFI).

Ambient interference was recorded using a data acquisition system with Lab View software and converted to the frequency domain by using a Fast Fourier Transform (FFT) algorithm. Most of the interference was found to be in the 60Hz to 10 KHz range and software filters built in to the LabView software were used to eliminate this interference. Other measures need to be taken to reduce noise and minimise the need for filters [5]. The total resistance of the circuit (film, wires and substrate) should be kept below 70 Ω; higher resistance leads to greater noise. Thermocouple wire pairs should be twisted around each other with grounded metal sheaths surrounding each pair to shield them. Voltage measurements should be done using differential mode sampling: the thermocouple wires should not be grounded and the ground must not carry the signal (as in common mode sampling). Selecting an effective ground is important, since the electrical grid creates significant noise: water pipes that go into the earth usually make good ground terminals.

To calibrate thermocouple junctions they were placed in a small furnace whose temperature could be controlled. A small enclosure, just large enough to contain the steel plate and thermocouple, was fashioned out of a castable refractory (Plycast AeroLite, RHI Canada Inc., Burlington, ON) and heated with a 400 W band heater. A temperature controller (CN9000A, Omega Engineering Inc., Stamford, CT) controlled the temperature of the furnace. Substrate temperature was measured with an accuracy of ±1°C using two K-type thermocouple attached to it. A multimeter (HP3468A, Hewlett-Packard, Palo-Alto CA) measured the thermoelectric voltage generated by the thermocouple and its reference junction with a resolution of 1 μV.

Calibration of the thermocouples was done by increasing substrate temperature from room temperature to 650°C in increments of 50°C. At each setting the surface temperature was allowed to reach steady state and the voltage generated by the thermocouple recorded. The surface was then allowed to cool with temperature decrements of 50°C and the voltage noted again. This process was repeated twice for each thermocouple. The results were reproducible within ± 9 μV, corresponding to an error of ± 0.2°C.

A fourth-order polynomial was fitted to each set of data. For a thermocouple junction of 303 stainless steel–Constantan the polynomial of best fit at temperatures between 20 and 600°C was:

$$T = -0.0658+27.157V-0.5212V^2+0.0147V^3-0.0002V^4 \quad (1)$$

where T is in °C and V is in mV. For a H13 tool steel – Constantan thermocouple, at temperatures between 20 and 650°C, the following expression can be used:

$$T = -1.156+22.635V-0.4135V^2+0.0105V^3-0.0001V^4 \quad (2)$$

Figure 4 shows these two polynomials graphically, compared to the output of a J-type (iron-Constantan) thermocouple. The output of all three thermocouples is similar.

4. Response time measurement

If radiant heat impinges on a thin film of thickness L, the time (t_r) for the thermal disturbance to propagate through the thickness of the film may be estimated, assuming one-dimensional transient heat conduction, by:

$$t_r \sim L^2/\alpha \tag{3}$$

where α is the thermal diffusivity of the film, with a value of approximately 1.5 x 10^{-5} m^2/s. Assuming L=0.5 μm, t_r is approximately 17 ns, which is extremely fast for a temperature sensor.

Control theory states that the dynamic performance of a sensor can be assessed by measuring its unit-impulse response-function (UIRF), defined as its response to a delta function $\delta(t)$ which has infinitely large amplitude, infinitely short duration and $\int_{-\infty}^{+\infty}\delta(t)\,dt = 1$. To test the thermal sensor a delta function can be approximated by a laser pulse of a few nanosecond duration. We therefore applied a single laser pulse on a surface of the thermal sensor while recording its output using a digital oscilloscope.

Figure 5 shows the experimental set-up. The thermal sensor was connected directly to a digital oscilloscope, without amplification or filtering A diode-pumped Nd:YAG laser was used to produce a single laser pulse with a duration of 15 ns and the response of the thermal sensor was recorded simultaneously by a digital oscilloscope (LECROY Waverunner LT354) with a maximum sampling rate of 1 GHz. Experiments were completed for two different laser pulse energies – 0.31 μJ and 0.46 μJ. This energy was high enough to erode the graphite film so that after a few pulses a new film had to be applied. Figure 6 shows two typical response functions of the thermocouple for 0.31 μJ and 0.46 μJ pulses.

The waveform of each UIRF has two separate phases, labeled "fast" response and "slow" response in Fig. 5. "Fast" response is the time period during which the sensor accumulates heat from the laser pulse and has at least one oscillation that corresponds to the second order dynamic system. Results from experiments showed that this response time is directly related to the thickness of the graphite film. New graphite films have longer response times, which decrease with subsequent pulses as the film erodes. "Slow" response is a time period when the sensor cools down as it dissipates heat by conduction to the substrate and finally reaches its equilibrium value. This dynamic behavior corresponds to the first order dynamic system. Therefore, the overall dynamic performance of the thermocouple is a combination of first and second order dynamic systems. Typical measured UIRFs (shown in Figure 7) have the following parameters:

- rise time of 4.66 ns and 5.38 ns,
- period of first oscillation of 35.66 ns and 34.38 ns, and
- amplitude of first oscillation of 13.13 mV and 22.03 mV

respectively for 0.31 μJ and 0.46 μJ pulses energies.

Thermocouple output voltages were converted to temperature by using equation (2). Figure 7 shows a typical variation of surface temperature with time after being exposed to a 0.46 μJ pulse. The nominal 15 ns duration of the laser pulse is also indicated.

5. Surface temperature variation measurement

Having confirmed that the thermocouple response was extremely rapid we tested it under more realistic conditions. Droplets of molten aluminium, 4 mm in diameter, were formed using a pneumatic droplet generator and allowed to fall under their own weight onto a H-13 tool steel plate from a height of 0.5 m so that the impact velocity was approximately 3 m/s. Details of the experimental apparatus have been given earlier [1,6]. The substrate had an average surface roughness of 0.5 μm and was mounted on a translation stage allowing it to be positioned precisely. The substrate had a line of sensors built on it, spaced 1.15 mm apart. The thermocouple at one end of the array was located exactly at the point of impact.

Thermocouple outputs were recorded using a data acquisition system (NI PCI-MIO-16XE-10, National Instruments Inc, Austin TX) with LabView software at a sampling rate of 10 KHz. Measurements were stored on a buffer in real time and downloaded to a computer off-line. Measured voltages were converted to temperatures using Eqn (2). Figure 8 shows the surface temperature history at five different radial locations under the splat. The first sensor to respond was that located directly at the point of impact (r=0). After delays corresponding to the time taken for the edge of the spreading droplet to contact each sensor in sequence, the temperature at each location increased very rapidly, to over 500°C in less than 0.1 ms, and then increased more gradually.

6. Summary and Conclusions

Thin film thermocouples have been developed to measure rapid surface temperature variation. The thermocouples can be built on any metallic substrate, are simple to build and rugged. They can be deposited on either smooth or polished surfaces and several sensors can be spaced close together. The dynamic performance of the thermocouple, represented by the on-line measured unit-impulse response function, exhibits complex dynamic behavior corresponding to a combination of first and second order dynamic systems. Tests showed that the thin film sensors can detect a 15 ns laser pulse. They have also been used to measure surface temperature variation under an impacting droplet of molten aluminium.

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Wire Material	Voltage output on a H13 Tool Steel Substrate	Voltage output on a 303 Stainless Steel Substrate
NiCr60	1.11 mV	0.84 mV
Copper	1.13 mV	0.77 mV
Iron	0.39 mV	0.28 mV
Constantan	10.45 mV	8.45 mV
Chromel	2.44 mV	4.86 mV
Alumel	4.54 mV	3.12 mV
Platinum	1.21 mV	1.08 mV

Table 1 Thermoelectric voltage differences generated at 200 deg C for different junctions

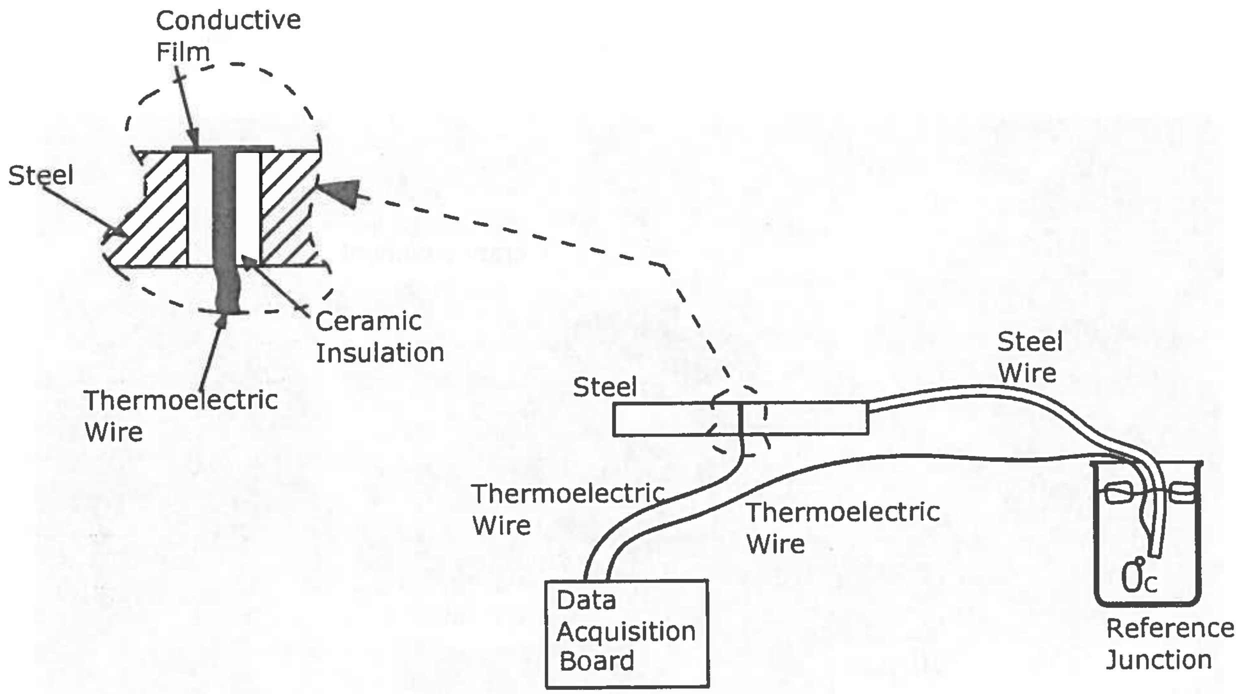


Figure 1 Schematic diagram of thin-film thermocouple.

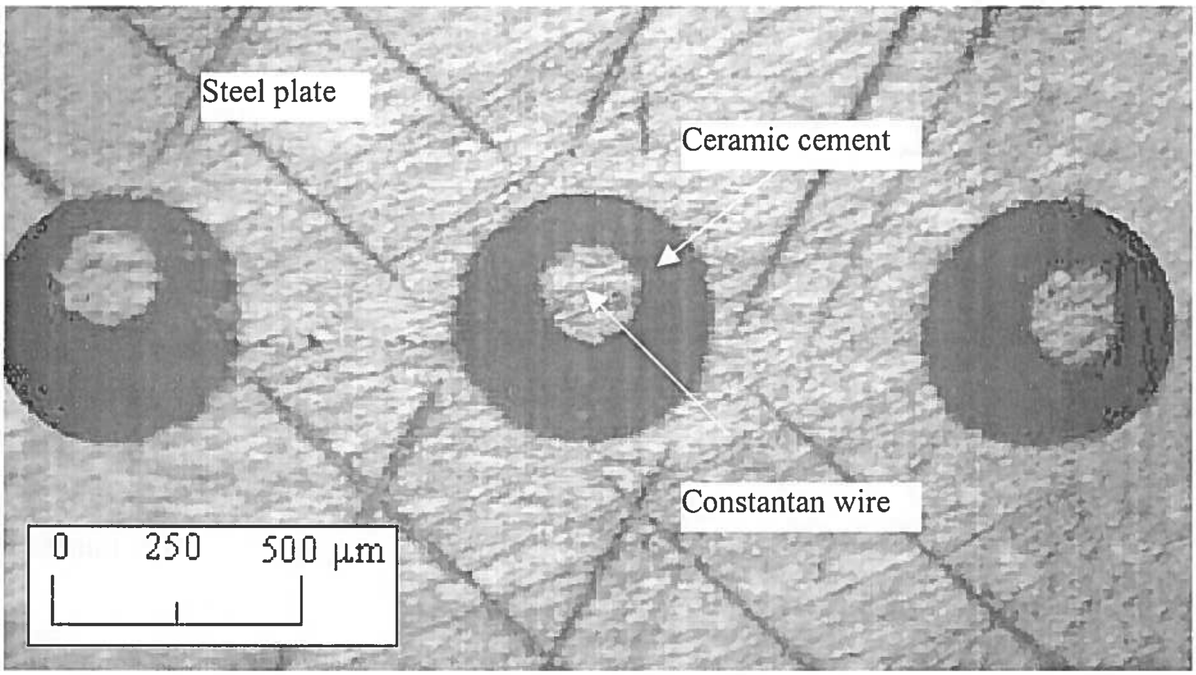


Figure 2 Top view of three temperature sensors placed 1.15 mm apart,.

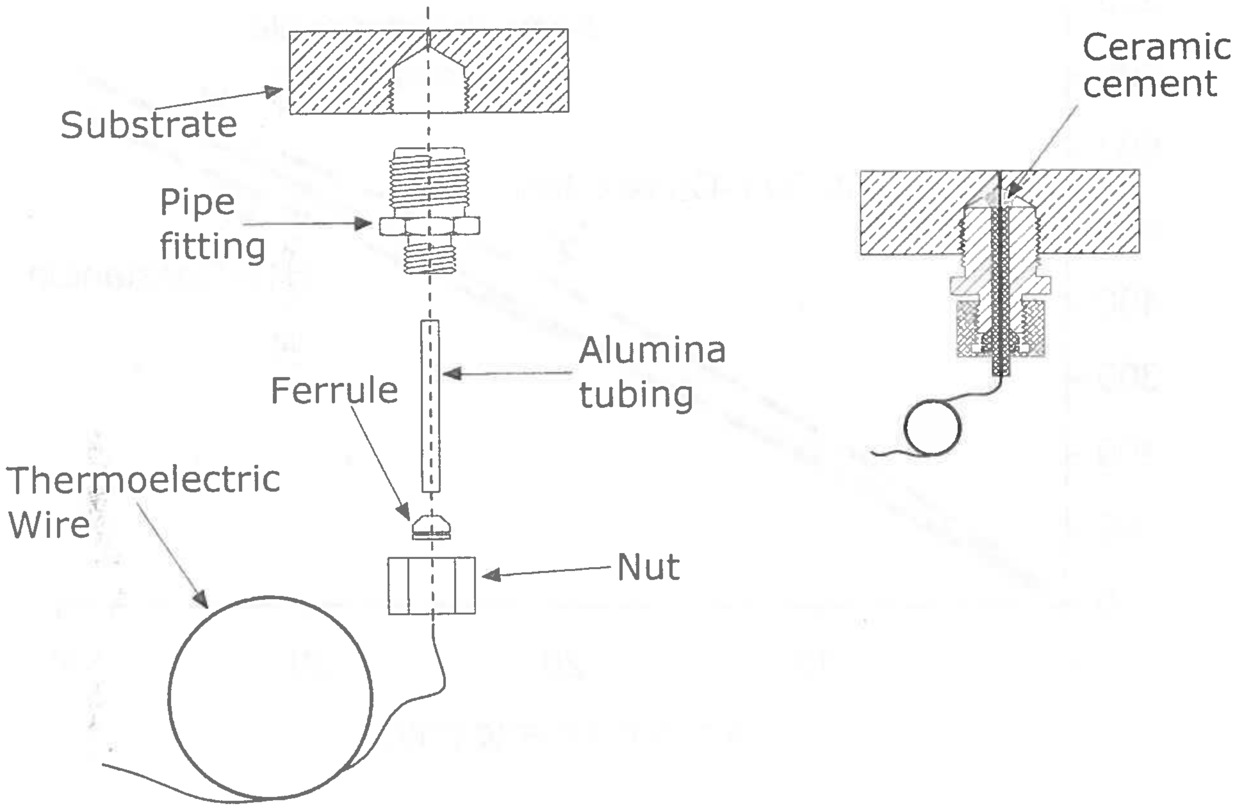


Figure 3 Thermocouple assembly

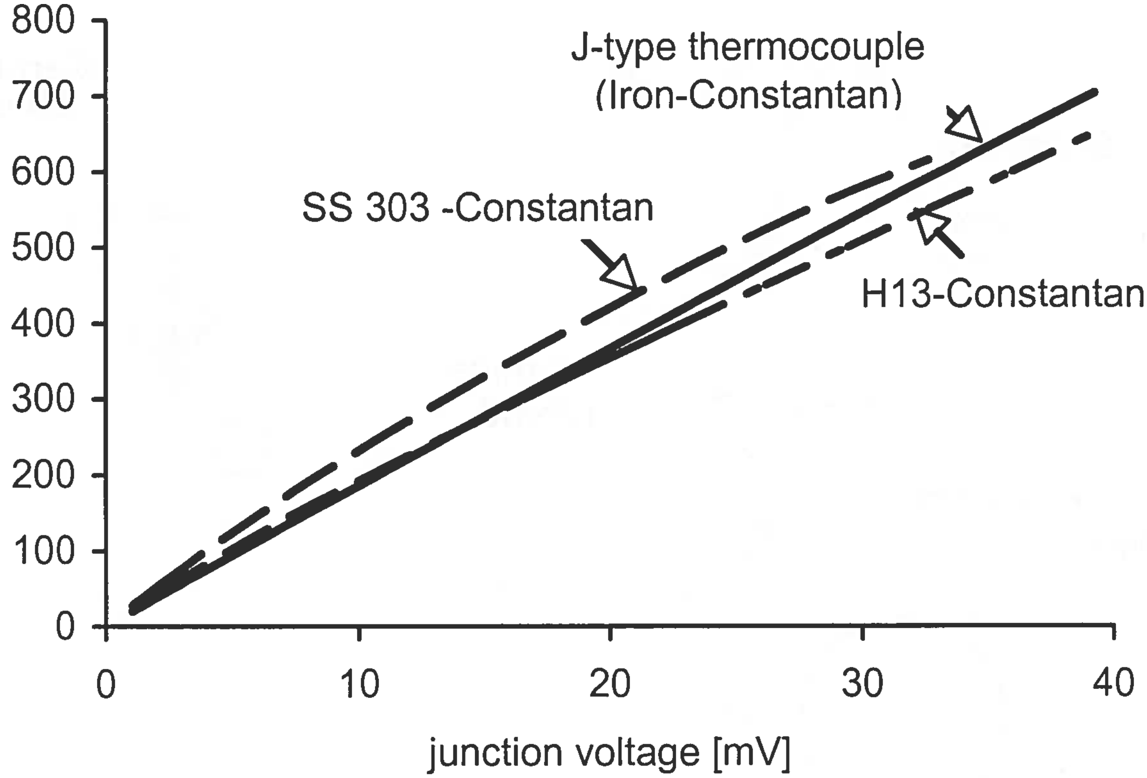


Figure 4 Variation of thermocouple voltage with junction temperature.

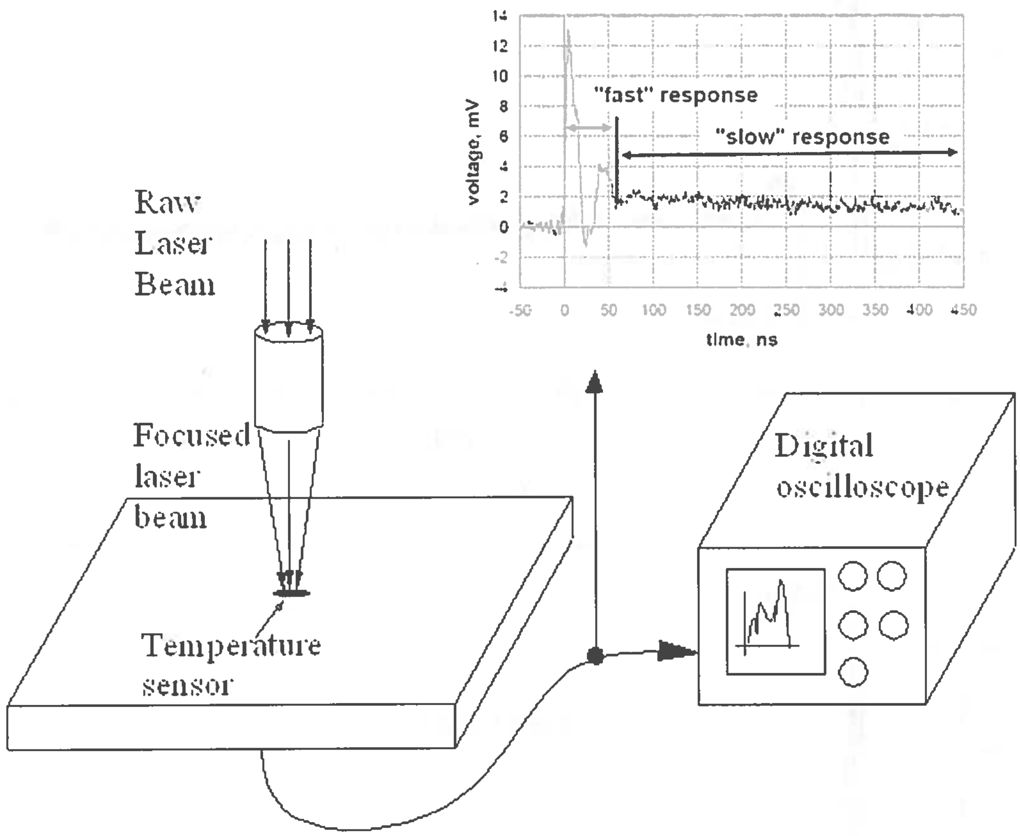
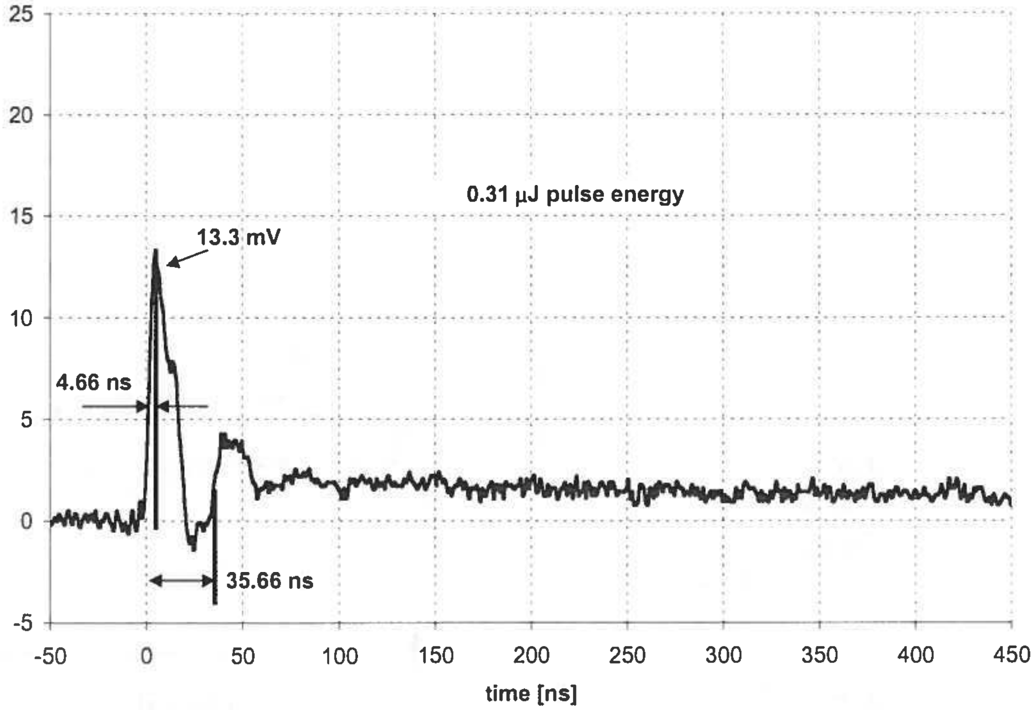
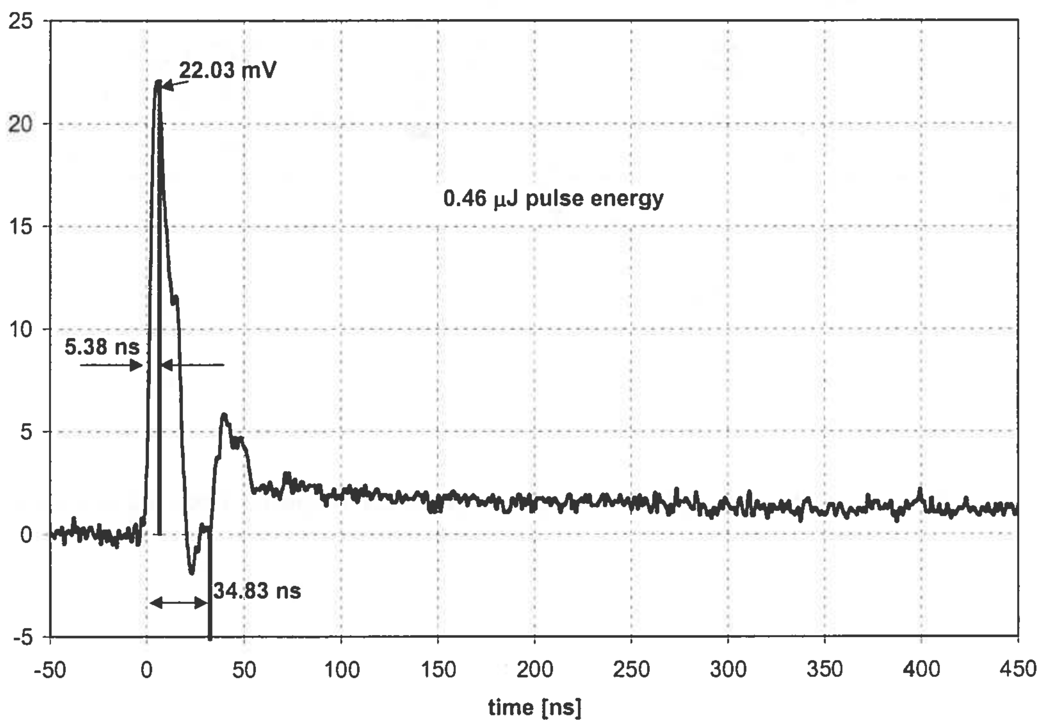


Figure 5 Schematic of experimental set-up for response time measurement.



(a)



(b)

Figure 6 Typical response functions of the thermocouple with a) 0.31 μJ pulse energy and b) 0.46 μJ pulse energy

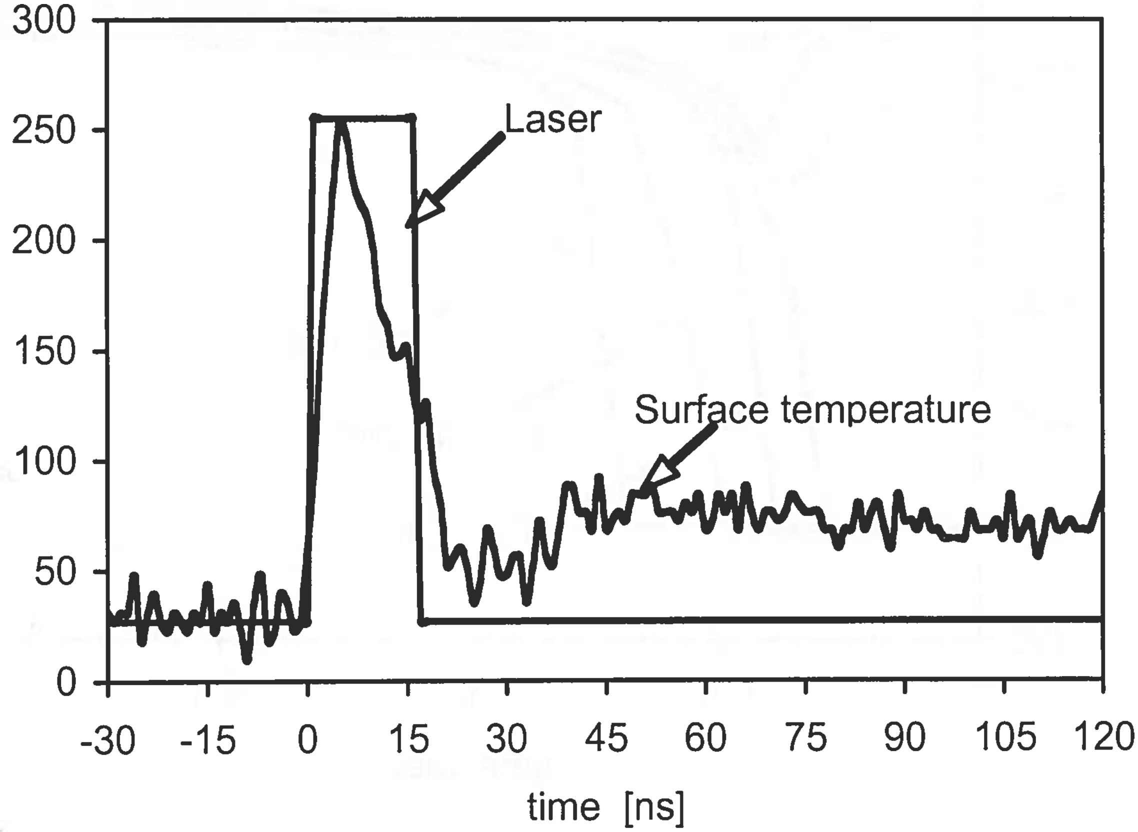


Figure 7 Thermocouple response to the application of a 46 μJ, 15 ns laser pulse applied directly to the thermocouple junction.

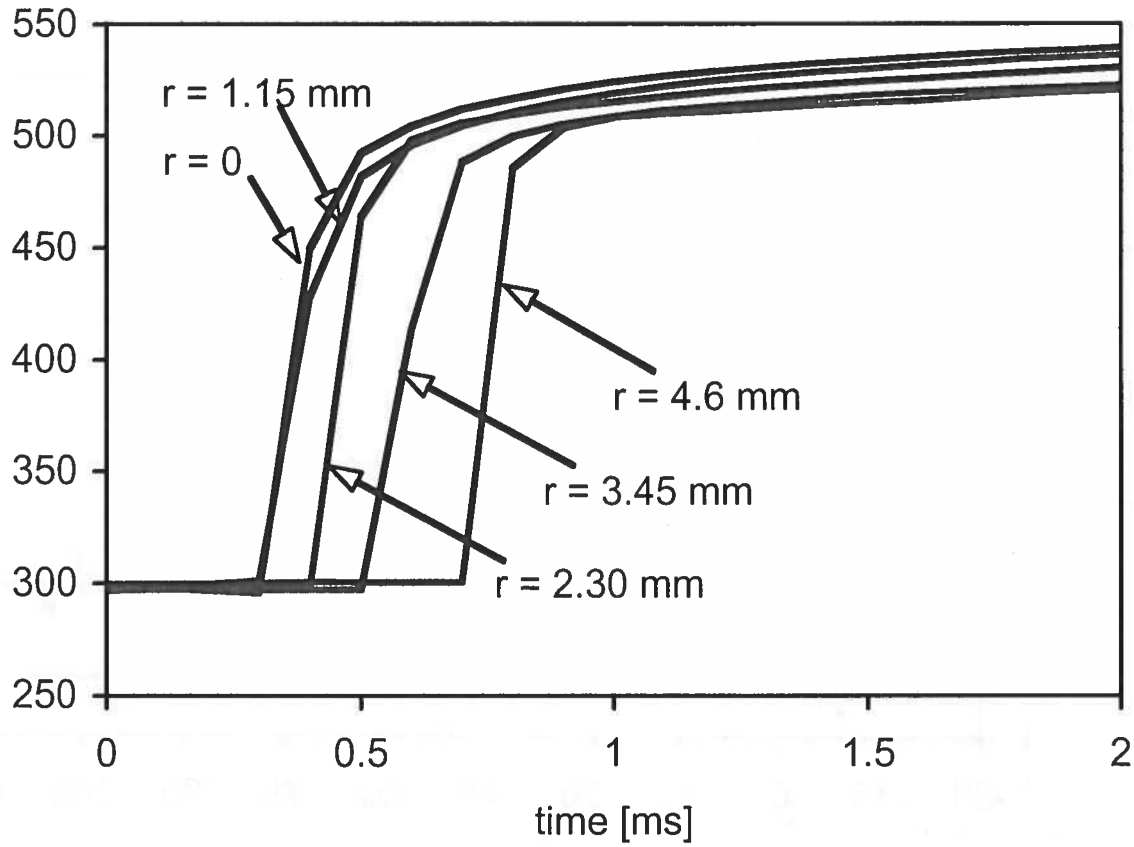


Figure 8 Response of 5 thermocouples located in a straight line at intervals of 1.15 mm from the point of impact of a 4 mm diameter molten aluminum droplet