COMPUTER-AIDED ENGINEERING DESIGN OF HTXRD RESISTIVE HEATING STRIPS TO MINIMIZE SAMPLE TEMPERATURE GRADIENTS

Rajeswari Chandrasekaran, Andrew R. Drews

Ford Research and Advanced Engineering, Ford Motor Company, Dearborn, MI 48124.

ABSTRACT
Reliable high-temperature XRD measurements require a uniform temperature distribution over the entire area of the sample. Typically, HTXRD stages use a resistive heating strip, where the ends that form the electrical current contacts are maintained near ambient temperature. Because of thermal conduction to the current contacts, the temperature distribution along the length of a short heating strip is roughly parabolic, leading to temperature gradients along the length of the sample. In addition to conductive losses, radiative and convective losses and temperature dependent properties such as electrical resistivity, emissivity and thermal conductivity also contribute to the temperature profile, making a direct analytical solution difficult. Although the roughly parabolic temperature profile of a heated strip is an unavoidable consequence of that geometry, there is no requirement that heating strips be uniform in their properties. Engineering the properties of a heating strip to minimize the temperature gradients over the sample area might be possible. We describe a theoretical and experimental study of two methods to flatten the temperature gradient in the center of a heating strip by introducing regions of high thermal and electrical resistances at the edges of the sample region.

INTRODUCTION
HTXRD is a useful tool to obtain information on crystallographic changes, phase transformations and other properties as a function of temperature. Precise determination of the temperature dependence of a material's properties requires a uniform temperature over the entire area of the sample. However, all HTXRD stages introduce temperature gradients that are reflected in a loss of precision, particularly in phase transitions (Fig.1).

Figure 1: (a) HTXRD scans showing the melting of aluminum metal. Note the existence of strong (311) texture that changes with temperature. (b) To avoid the ambiguity introduced by texture changes, the averaged background in the vicinity of 40 degrees is used to determine the melting transition temperature and width.

HTXRD stages that use a simple resistive heating strip make electrical current contacts at the ends of the strip and maintain those ends near ambient temperature (typically water cooled).
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An example is shown in Fig. 2. Because of thermal conduction to the current contacts, a
temperature gradient along the length of the strip is established that is roughly parabolic. In addition to conduction to the ends of the strip, convection of heated gas and radiation can be important loss mechanisms and will influence the temperature distribution. In particular, radiative loss has a very strong power-law dependence on temperature \(T^4\) and tends to flatten the temperature distribution, but this effect is inefficient except at fairly high temperatures. Some manufacturers of HTXRD chambers have attempted to mitigate thermal gradients at the sample region by providing a secondary "surround" heater, though the effectiveness of this approach has been questioned.

Figure 2: (a) Buehler HDK HTXRD furnace assembly showing the heating strip, sample, surround heater and current contacts (b) Schematic of heater configuration.

With the inclusion of convective and radiative losses in the energy balance describing the heat flows along the heater strip as well as temperature dependent material properties, analytical solutions are not possible and accurately modeling the temperature distribution along a strip requires numerical approaches. Using a modern multi-physics finite-element method, two alternate designs to flatten the temperature distribution are investigated here along with experimental verification. These approaches alter the electrical and thermal resistances of the basic strip heater as a function of position to control both the heat source and loss terms in the energy balance of the strip. This is achieved by: (a) introducing regions of high thermal and electrical resistances at the edges of the sample region by drilling holes, (b) varying the thickness of the strip along its length. Modeling and simulations are an effective method to reduce the time required to analyze the effects of changing materials, geometry (design), configuration and temperature (with and without surround heater) and are helpful in guiding the optimization.

THEORY
For simplicity, we begin with the simple resistive heating strip given in Figure 3. In this case, the electrical current flows along its length and heat is generated ohmically. Moreover, the heating strip has a finite thermal conductivity, which can be a function of temperature. Hence, the net rate of change of temperature in each differential element is a combination of the ohmic heating and the conductive heat flux in and out of the element. At steady state, the energy balance in the strip is described as

\[
\nabla \cdot \left( k_t(T) \nabla T \right) = \frac{i^2}{k_e(T)}
\]  

(1)
where $k_t(T)$ and $k_e(T)$ are the temperature-dependent thermal and electrical conductivities and $i$ is the electrical current density.

*Figure 3: Baseline heating strip design showing the position of the sample area between the ends ($x=0, L$) where the temperature was fixed at 298K.*

The electrical current density $i$ can be related to voltage drop $V$ across the total strip length $L$ (i.e. along the x-axis):

$$i = \left( k_e(T) \right) \frac{V}{L}.$$  \hspace{1cm} (2)

Substituting (2) in (1), we obtain

$$\nabla \left( -(k_t(T)) \nabla T \right) = \left( k_e(T) \right) \left( \frac{V}{L} \right)^2.$$ \hspace{1cm} (3)

In this work, an iron strip was used for the baseline case and its material properties are used in the simulations. The two ends (yz-planes) at $x=0$ and $x=L$ are held at 0 and $V$ volts respectively and both held at ambient temperature (298K). A surround heater is usually used to minimize thermal gradients in the sample area and we have modeled its effect by assuming that it limits heat flow out of the other exposed faces in the same fashion as thermal insulation. Therefore, when considering the effect of a surround heater, we assume a boundary condition where the outward normal heat flux from these faces is zero. If there are no surround heaters, then there will be convective and radiative heat losses from these faces of the strip and are given as

$$-(k_t(T)) \nabla T = h (T - T_{ext}) + \varepsilon(T) \sigma (T^4 - T_{ext}^4)$$ \hspace{1cm} (4)

where $\varepsilon(T)$ is the emissivity of iron as a function of temperature, $\sigma$ is the Stefan-Boltzmann constant, $(5.6703 \times 10^{-8} \text{ W/m}^2/\text{K}^4)$ and $h$ is the convective heat transfer coefficient, W/m$^2$/K.

**DESIGN APPROACHES**

Empirically, the inadequacies of the simple heater strip (with ends at ambient temperature) can be summarized as: 1) The relative magnitude of the temperature gradients in the sample area vary considerably with temperature, showing a minimum when sample temperature is near ambient or at very high temperatures, but a maximum at intermediate temperatures where most experiments are conducted (for the same convective heat transfer coefficient); 2) too little heat is generated just outside the sample area, resulting in a temperature profile that is "peaked" in the middle. Although the alternate designs that we consider are mainly focused on accomplishing a solution to the second concern, designs are also judged by how well they mitigate the first inadequacy.
In Figure 4a we compare the parabolic temperature profile from a simple strip with no convective or radiative losses to a hypothetical ideal case. From this comparison, it is clear that the temperature gradient across the sample area is either the result of too little heat produced at the edges of the sample zone, or as too much heat produced in the center of the sample zone. In Figures 4b and 4c, we illustrate two approaches: increasing heating just outside the sample zone by reducing the amount of conductor by drilling holes through the heater strip (4b), or, adding a thick conductive pad in the sample area ("platform design") that effectively short-circuits the heater in the sample zone and thereby increasing the relative amount of heat generated outside the sample zone (4c).

Figure 4: (a) A comparison of the temperature gradients in the central zone in a simple heater strip with no convective or radiative losses (red) and in an ideal design. (b) A design with holes drilled through the strip outside the sample area intended to increase the relative amount of heating in these regions. (c) A design that uses a thick platform welded to the strip in the sample area to decrease the heating in the central zone and thus, enhance the relative amount of heating outside the sample area. Both these designs aim to flatten the temperature distribution over the sample area.

Both designs have been simulated and also experimentally tested using infrared imaging to directly measure the temperature distribution. In some cases, the effectiveness of the approach was also tested using the melting transition of aluminum. The details of these experiments are given below. For the multiphysics simulation, the baseline strip design was taken to be a 40mm long × 8mm wide × 0.5mm thick strip (similar to experimental set-up) and the convective heat transfer coefficient is assumed to be 10 W/m²/K for all simulations.

**SIMULATION IN COMSOL MULTIPHYSICS SOFTWARE**

All simulations are carried out using COMSOL Multiphysics software version 4.2 which uses the finite element method. The Joule heating physics interface, with modifications to reflect the boundary conditions described in theory and desired material properties, is used. Stationary study (and hence appropriate stationary solvers) are chosen for the steady-state simulations in this work. Once the desired geometry is created, the free tetrahedral meshing option is used and refinements in the distribution and number of node points along edges are made as necessary to optimize between the computational time (for the same computing power) and accuracy.

**EXPERIMENTAL SET-UP**

Temperature gradients in the sample area of a strip heater were determined by both HTXRD measurements of the melting transition of aluminum and by infrared thermography. The sample
for the aluminum melting was a fine metal powder dispersed onto a SrTiO₃ (100) crystal. The crystal was located in the center of the heating strip and diffraction data were collected using Cu K radiation and in a chamber flushed with nitrogen. Scattered radiation was measured with a Inel CPS 120 position sensitive detector that covered a 120 degree arc. During the experiment, diffraction intensities from the Al fcc lattice showed considerable preferred orientation that changed during the course of the experiment (Fig. 1). Data were collected at a series of temperature steps that spanned the melting point. At each step, the temperature was stabilized for several minutes before diffraction data were collected. The continuous changes in texture made estimating the melting point from the disappearance of the diffraction peaks difficult and instead, the melting transition was determined from the sudden appearance of an amorphous background in the vicinity of 35 to 45 degrees two-theta. To reduce the effect of noise, the background was averaged over the range of angles where the amorphous “hump” appears. The averaged intensities were then fitted with a sigmoid curve to estimate the temperature offset and width of the transition due to the temperature gradients across the sample area. IR thermography was used to measure the thermal gradients over the surface of the heater strip, including areas outside of the sample area using an FLIR Indigo camera equipped with optics that transmit in the 3-5 micron band. To prevent saturating the image sensor, neutral density attenuators were improvised from sheets of glass and acrylic. Raw image data were enhanced and color coded using the NIH ImageJ program and line profiles calculated.

DESIGN RESULTS AND VALIDATION

The simulated temperature distribution on the strip for the baseline design with ends held at 298K and radiation and convection losses is given in Figure 5a for a potential difference of 0.42 V. The temperature profiles for 3V, 0.43 V and for 0.42 V with or without thermal insulation (simulating the presence and absence of surround heater (SH) respectively) on the four faces other than the two ends at 298K are also provided in Figure 5b. Within the area of the sample (central 10 mm) the temperature varies by as much as ~ 100K at a maximum sample temperature of 1000K. At very high temperatures, radiative and convective losses tend to flatten the temperature profile. Ideally, the temperature profile in the sample area would be flat for a wide range of temperatures that are appropriate for most HTXRD experiments. This suggests that alternate heater strip designs must be considered.

**Figure 5:** (a) Simulated temperature distribution in the baseline heating strip design. (b) The temperature profile is shown for several applied voltages and illustrates the variability in the temperature gradient across the sample area with desired HTXRD temperature. (c) An IR image is shown for a similar heating strip with a central temperature of 300°C. (d) The uncalibrated temperature profile was derived from (c) by averaging the pixel value vertically for each horizontal position.
For a simulated case where the voltage across the strip is held constant at 0.42V, adding a surround heater raises the central temperature of the strip by ~ 200K, which is roughly equivalent to increasing the voltage across the strip to 0.43V without a surround heater. However, there is very little apparent difference in the temperature gradients across the sample area between these two cases, suggesting that its effectiveness is limited for this design and at this temperature.

**ALTERNATE DESIGNS:**

**Case 1-Heating strip with holes:** The temperature profiles for the design in Figure 4b with holes added to increase the strip’s electrical resistance outside the sample area are investigated at several different voltages and are shown in Figure 6. The temperature profile in Figure 6a tends to flatten along the length of the strip and reaches the desired temperature range ~ 1100K at the center of the strip. However, the temperature gradient at the center of the strip over a distance of 10 mm is still ~ 50 K. If the surround heater provides sufficient thermal insulation on the exposed four sides (except the ones at 298K) such that losses can be neglected, for the same potential difference, the maximum temperature and also the temperature gradient are higher. However, as the potential difference is further increased to 1 V (with radiative and convective losses included), the temperature profile flattens nearly completely. At higher voltages and hence temperatures, the gradient increases again, though now in the opposite sense.

![Figure 6: (a) Simulated temperature profiles and distribution (inset) for the heating strip with holes positioned as shown in Fig. 4b. The magenta and blue curves are for the same voltage, with and without a surround heater, respectively. The cyan and purple curves are shown for no surround heater. (b) An IR photo of a heating strip with drilled holes and (c) the corresponding temperature profile.](image)

Although the design with holes shows a nearly flat temperature profile at ~1600K, such high temperatures are not typical in HTXRD and curling or even melting of the strip can occur. Preliminary simulation results indicate that with an optimal number and arrangement of holes, the temperature profile at the center of the strip can be flattened at ~ 1100 K. The practicality of this design must be evaluated in future experiments.

**Case -2: Heating strip with varying thickness ("platform" design):** An alternate design featuring a short, thicker (800 μm) strip bonded onto a longer, thinner (50μm) foil (shown in Fig. 4c) is also investigated. In this design, the thicker section effectively provides an electrical short-circuit across the sample area, limiting the heat produced in that region and confining the majority of
the heating to areas outside the sample area. The simulated temperature profiles at several 
temperatures are shown in Figure 7 and demonstrate that the thicker strip does flatten the 
temperature profile at the center of the strip, though it leads to high temperature "ears" just 
outside the sample area for a platform thickness of 800μm. This effect is validated in the IR 
thermograph (Fig. 7b) and line profile (Fig. 7c). To reduce this effect, a further variation on the 
design is tested where the center strip thickness is reduced to 200μm. At all temperatures, the 
"ears" are much less evident and a flattening of the temperature profile in the central sample area 
is preserved over a wide temperature range, especially in the presence of surround heater.

**Figure 7:** (a) Simulated temperature profiles for the “platform” heater design at three different 
temperatures (voltages) and for various thicknesses of the platform, with (SH) or without (nSH) a 
surround heater. A curve designated “800 nSH” implies a 800μm platform on a 50μm foil without a 
surround heater. The false-color IR image of a platform heater at 300°C is shown in (b) with the 
corresponding temperature profile plot is shown in (c).

**CONCLUSIONS**

Computer aided design of heating strips for HTXRD to minimize temperature gradients across 
the area of the sample has been demonstrated and validated with preliminary experimental 
results. The effects of adding a surround heater is shown not to offer much benefit in reducing 
the temperature gradients along the length of the baseline strip heater. Adding a high-resistance 
region just outside the central sample area by drilling holes in the heating strip does reduce 
temperature gradients in the sample area, though it does not appear that this design satisfies the 
need for a flat temperature profile over a wide temperature range and at temperatures normally 
used in HTXRD measurements. A further variation where a thicker central platform is bonded to 
the strip has been shown to reduce gradients more effectively and over a wider range of 
temperatures, especially when the thickness is optimized and radiative and convective losses are 
avoided. Further validation experiments are planned. The utility of multiphysics simulation 
methods to investigate complex geometries and several conditions has been clearly demonstrated 
and the method is an efficient and accurate approach to pre-screening and optimizing designs.

**REFERENCES**

1. H. Wang, E. A. Payzant, "Infrared Imaging of Temperature Distribution in a High Temperature X-ray 