Design of a micromanipulation system for high temperature operation in an environmental scanning electron microscope (ESEM)

P Samara-Ratna\(^1\), H V Atkinson\(^2\), T Stevenson\(^1\), S V Hainsworth\(^2\) and J Sykes\(^1\)

\(^1\) Space Research Centre, Department of Physics and Astronomy, University of Leicester, University Road, Leicester LE1 7RH, UK
\(^2\) Department of Engineering, University of Leicester, University Road, Leicester LE1 7RH, UK

E-mail: hva2@le.ac.uk

Received 4 September 2006, in final form 13 November 2006
Published 5 December 2006
Online at stacks.iop.org/JMM/17/104

Abstract

The environmental scanning electron microscope (ESEM) allows the sample to be imaged under a low pressure atmosphere. The ability to micromanipulate with precision within heated power systems in an ESEM will enable a greater understanding of the behaviour of materials at high temperature. Heating stages for ESEMs are commercially available but none include micromanipulation systems. Creating such a system is fraught with design problems. This is because the piezoelectric transducers, required to generate the precise range of movement within the heated environment, are unable to operate at temperatures exceeding about 90 \(\degree\)C and require thermal protection. Here, we have used a one-dimensional probe as a model of a three-dimensional manipulator. We have introduced a thermal break and a thermally insulating extension arm to protect the piezoelectric from heat. We have applied finite element analysis to test the design concepts before practical implementation. This ensures that the piezoelectric transducers in the costly practical devices are not placed at risk. The predictions have been validated with subsequent experimental work although there are some discrepancies to resolve. Examples of the movement of aluminium alloy grains and copper powder particles using the one-dimensional manipulator are given.

1. Introduction

An environmental scanning electron microscope (ESEM), in contrast to a traditional scanning electron microscope which operates under high vacuum, allows a sample to be imaged under a low pressure atmosphere. Common atmospheres used in an ESEM include water vapour, argon or nitrogen \(^1\). An electron beam ionizes the gas to give positive ions and these, in turn, reduce the charge build-up on the material being sampled, which would otherwise charge negatively due to bombardment with electrons. The ionized gas also acts to amplify the image signal when secondary electrons are used to image the surface. The ESEM can be used with a heating stage where the sample is placed in a small ceramic crucible located above a heater. Above 600 \(\degree\)C, a heat shield, with a small hole to allow imaging, is placed above the ceramic crucible to protect the detector from heating effects. The gaseous secondary electron detector (GSED) is situated directly above the sample, around the pole piece, and is used to acquire images when the heating stage is in use; other detectors are sensitive to the light emitted by hot samples. The ESEM is, thus, a powerful tool for observing the behaviour...
of materials at high temperatures. Heating stages for the ESEM are available commercially but there is no such system that would allow the micromanipulation of materials whilst at these high temperatures. This would be valuable, for example, for obtaining detailed micromechanical information on microstructure evolution during sintering, including under applied stress [2]. Micromanipulation systems are well established for use under ambient conditions. The difficulty at high temperatures is that the piezoelectric transducers, used to generate the precise range of movement, begin to permanently degrade by depolarization above a certain temperature. The manufacturers of the devices we are using recommend not operating above about 90 °C. There is, therefore, an engineering design challenge in establishing a system for protecting the piezoelectric crystals from the heat. This challenge is increased by the restrictions introduced by operating within the space inside the ESEM chamber, in particular the space between the heating stage, the heat shield and the GSED. The aim here was to use finite element analysis to predict the heat flows for design concepts so as to ensure acceptable conditions for the piezoelectric device during operation. The predictions were then validated through experiment. The system established is the first high temperature micromanipulation system in an ESEM. It offers the potential not only for experiments which involve heating stages for the GSED of the ESEM. During imaging, the GSED is very close to the heat shield. A small hole in the heat shield allows the centre of the crucible to be viewed. The heating is controlled by a dedicated controller, with an LCD display, which allows the setting of target temperature, temperature ramp and cooling rates. The temperature of the crucible is measured using a thermocouple located directly beneath the heating coil. There may be some discrepancy between the displayed temperature values and the actual value because of the separation between the thermocouple and the crucible. The manufacturer (see footnote 3) states that final calibration is performed with the heating stage installed inside the ESEM and the temperature reading must fall within ±20 °C of the calibration temperature (at 760 °C) before the stage is accepted to shipment. Calibration is achieved with the use of calibration paints which visibly change at a specified temperature. The position of the thermocouple is adjusted until the composition of the paint changes within the required temperature range.

For the work reported here, the design concept was initially based on a one-dimensional micromanipulator available from PiezoMotor®. This provides movement in one axis in steps of ~5 μm. The use of a one-dimensional manipulator allowed ideas to be tested with a relatively inexpensive piece of equipment, as opposed to using three-dimensional manipulators available in the market. Once 1D manipulation has been proven the principle will be extended to 3D manipulation. To protect the piezoelectric device from heat a probe extension arm made of an insulating material (in this case, a hollow alumina tube) was introduced. The manipulator was mounted to one side of the heating stage within an aluminium frame (figure 2). The 1D probe (11 in figure 1 and 4 in figure 2 and 2 in figure 3) was not mounted on the heating stage because it needs to move independently from the three axes of movement of the stage itself. A copper block (3 in figure 2 and 4 in figure 3) was introduced between the alumina arm of the piezoelectric motor and the probe arm to create a thermal resistance. It also acts as a heat sink

### 2. The heating stage and the design concept

The ESEM used in this work is a Philips XL30 ESEM with a heating stage supplied by FEI (see footnote 3) (figure 1). The heating stage heats the alumina crucible (diameter 5 mm) containing the sample to temperatures between 20 and 1000 °C. The heat is generated by a wire coil located beneath the crucible. The heater wire is surrounded with a ceramic adhesive and potted into an alumina holder. The alumina is surrounded by an insulating zirconia-based ceramic called Zircar ZYZ-3. This is separated into two separate sections to allow the alumina holder to be installed and is encased in a water-cooled aluminium housing. A stainless steel heat shield is located above the crucible and provides thermal protection for the GSED of the ESEM. During imaging, the GSED is very close to the heat shield. A small hole in the heat shield allows the centre of the crucible to be viewed. The heating is controlled by a dedicated controller, with an LCD display, which allows the setting of target temperature, temperature ramp and cooling rates. The temperature of the crucible is measured using a thermocouple located directly beneath the heating coil. There may be some discrepancy between the displayed temperature values and the actual value because of the separation between the thermocouple and the crucible. The manufacturer (see footnote 3) states that final calibration is performed with the heating stage installed inside the ESEM and the temperature reading must fall within ±20 °C of the calibration temperature (at 760 °C) before the stage is accepted to shipment. Calibration is achieved with the use of calibration paints which visibly change at a specified temperature. The position of the thermocouple is adjusted until the composition of the paint changes within the required temperature range.

For the work reported here, the design concept was initially based on a one-dimensional micromanipulator available from PiezoMotor®. This provides movement in one axis in steps of ~5 μm. The use of a one-dimensional manipulator allowed ideas to be tested with a relatively inexpensive piece of equipment, as opposed to using three-dimensional manipulators available in the market. Once 1D manipulation has been proven the principle will be extended to 3D manipulation. To protect the piezoelectric device from heat a probe extension arm made of an insulating material (in this case, a hollow alumina tube) was introduced. The manipulator was mounted to one side of the heating stage within an aluminium frame (figure 2). The 1D probe (11 in figure 1 and 4 in figure 2 and 2 in figure 3) was not mounted on the heating stage because it needs to move independently from the three axes of movement of the stage itself. A copper block (3 in figure 2 and 4 in figure 3) was introduced between the alumina arm of the piezoelectric motor and the probe arm to create a thermal resistance. It also acts as a heat sink

3. FEI Company, 5350 NE Dawson Creek Drive, Hillsboro, Oregon 97124 (www.fei.com).
4. Zircar Zirconia, Inc., PO Box 287, Florida, NY 10921-0287 (www.zircarzirconia.com).
5. Piezomotor Uppsala AB, Sylveniusgatan 5D, SE-754 50 Uppsala, Sweden (www.piezomotor.se).
but the material was not chosen to optimize this (otherwise a higher emissivity would have been sought). Minimizing heat transfer from the copper block to the temperature sensitive piezoelectric motor was achieved by connecting the square-section piece of alumina on the piezoelectric motor into a drilled cylindrical hole in the copper block such that a friction fit is formed as shown in figure 2.

In this arrangement the conduction of heat to the piezoelectric motor was only possible through the edges of the alumina and the smooth, shiny surface of the copper block limits the radiative heat transfer. The probe arm was sufficiently small to fit through the narrow gap of approximately 2 mm created between the heat shield and the stainless steel cover of the heating stage. At the end of the probe, a tungsten tip was attached which juts down into the ceramic crucible and, hence, into the sample to provide manipulation (see figure 3). The axis of movement was parallel with the probe arm. A vertical height adjuster was required to ensure that the manipulator arm was at precisely the right level to fit under the heat shield without lifting it. This was part of the aluminium mounting frame. A temperature sensor was attached to the copper block as a precaution to ensure that the piezoelectric motor was not damaged.

3. Construction of the finite element model

A computed-aided design (CAD) model of the heating stage was created using dimensions provided by the heating stage manufacturers, FEI (see footnote 3). The model was appropriately meshed to create the finite element (FE) model shown in figure 4. All modelling was performed using the IDeas engineering software [3] in conjunction with MAYA’s TMG solver [4]. Components of the heating stage were modelled as accurately as possible and included modelling of the heater wire coils and cooling fluid. The high level of detail was introduced as initially it was not clear which features of the heating stage dominated performance. The final model contained 10281 nodes and 22216 elements. Mesh density was increased in the locality of the heat source to improve results. Other components outside the area of prime interest were meshed with the largest possible mesh to reduce computation time.

In these experiments, the ESEM was operating at pressures of 2 Torr or lower. Taking into account the pumping speed and ESEM chamber size, the flow conditions are considered to be transitional and molecule mean free path is close to the chamber diameter [5]. In these conditions, the primary modes of heat transfer for the heating stage are by conduction and radiation. Therefore, convection is considered negligible and not included in the analysis.

Conductance modelling was performed using the finite element centre of gravity (CG) method. This method establishes a calculation point in the element’s centre of gravity as well as at each boundary [6]. Conductance is established between the boundary points using an algorithm which constrains linear element temperature functions to...
satisfy the governing partial differentiation equation. The calculation point at the centre of gravity is used to distribute heat transfer to the boundary nodes in the element. The element temperature is computed assuming a piecewise linear temperature distribution in the element. The solver evaluates temperature at each calculation point and once the matrix is solved temperatures are interpolated to the mesh nodes. The CG method is chosen in preference to the element centre method as it generates high accuracy results, facilitates modelling of temperature-dependent properties and has little sensitivity to element distortion. However, this led to longer solve times.

Each component of the heating stage is filled with linear tetrahedron solid elements and covered in a shell of null thickness. The conductance is achieved by defining thermal coupling between null shell layers. This method enables heat transfer to occur between bodies without the need for a physical coupling. For analysis, conductance paths between materials in close proximity are assumed perfect. This is due to the difficulty in determining the degree of contact between parts. This assumption may be the cause of errors as without contact only radiative heat transfer occurs. However, it is extremely difficult to determine an appropriate conductance value as many variables are involved. The heating stage was disassembled and it was found there was near-friction fit between parts and this gave confidence that a perfect contact assumption could be applied. Perfect contact is simulated in the model by specifying very high conductance values in the thermal coupling between parts.

Linking different parts by thermal couplings must be performed carefully to avoid production of thermal ‘short circuits’. If thermal couplings are applied incorrectly the model can produce unnatural conductance paths which produce unrealistic results. Validation of the model was achieved by applying thermal boundaries around groups of parts with thermal couplings and comparing the results to hand calculations using Fourier’s conductance law. This method of validation allows gross errors in the model to be identified without the need to generate complex validation calculations. However, this method does not allow small errors to be identified as agreement between hand calculations and the analytical model is often not close. In most cases, the user had to determine whether the results were sufficiently close for acceptability, based on the area of the model being considered. For example, the walls of the alumina holder (labelled as 4 in figure 1(b)) surrounding were checked by applying a 1000 W heat load on the inside surface while the external surface is held at 250 °C. Fourier’s law determines that the interior wall temperature should be 1067 °C and the model predicts 932 °C, thereby giving a 12.7% error. This is sufficiently close given that the calculation does not account for the thicker bottom section of the alumina holder. The thicker bottom section will cause the calculated result to be higher than the FE model. This gives further confidence that this region of the model operates correctly.

Distorted elements can result in the generation of negative conductances within elements. The mesh was checked for angles larger than 135° as these can cause potential conductance problems [7]. Where these elements existed, the mesh density was redefined until as many large angle elements as possible were removed from the analysis. Removal of all large angles was not possible and the solver was required to perform some modifications to conductances within elements. Steps were taken to ensure that conduction modification was minimized and ultimately only 0.44% of the elements were affected. The modified elements were not constrained to a single part and were distributed throughout the model. Therefore, the conduction modification was considered sufficiently small to be ignored and results would be dominated by the correctly operating elements.

I-Deas TMG thermal analysis (TMG) allows the thermal couplings between elements to occur either between elements that overlap across the thermal coupling or between all elements. In all cases, it was appropriate for heat transfer to occur between overlapping elements. Generally, it was discovered that connecting thermal couplings using all available elements resulted in an unnatural dispersion of heat through the system as unrealistic thermal paths were generated.

Radiation is modelled using the standard radiation solver within the TMG package. Heat transfer between internal components of the heating stage is dominated by conduction as contact is assumed perfect. Therefore, radiation modelling was only applied to surfaces exposed to ambient conditions and was only performed between the thin shells surrounding solid parts and beam elements. Solid elements are ignored and reliance is placed on the null shells surrounding the parts to generate radiative heat transfer. To absorb energy from the system, a ‘space enclosure’ is created around the stage. The space enclosure creates one large cube made of six or more elements that enclose the model and local space around it [7]. The calculation of radiative heat transfer is based on view factors between pairs of elements. Objects with direct line of sight have their view factors calculated first. The remaining elements have shadowing checks performed to determine the proportion of radiative heat transfer available based on unobstructed area [7].
### 4. Material properties

The thermal properties of materials were carefully chosen and defined to vary with temperature to produce realistic results. Most properties were extracted from standard textbooks and from manufacturer data sheets. However, emissivity and absorptivity are dependent upon surface finish and environmental conditions and these properties were varied to see what effect the changes had on the overall results and used as a means of fine tuning the model. The final material properties used in the model are given in Table 1. The sensitivity of the FE model to the values is evaluated by changing, in turn, all thermal conductivity values by 50% from the optimal value and determining the percentage change of temperature at the centre of the crucible. The dominance of the thermal conductance of the Zircar is clear as the sensitivity to its thermal conductivity is approximately 22 times greater than that of the next most influential material alumina. The table shows conductance values up to 3000 °C and this is above the maximum operable temperature for many materials. However, the values were used as an upper limit in calculations and in analysis temperatures were never developed in this region.

### 5. Results from the FE model for the heating stage

Heat is generated in the model by applying a transient heat load to the wire coils of the heater. The heat load is derived from experiments in which voltage and current measurements were taken during the heating cycles as shown in Figure 5.

In a full analysis on a standard Mobile Pentium Laptop computation time for a full transient analysis takes approximately 1 h.

The FE model shows that the performance of the heating stage is dominated by the thermal conductivity of the Zircar surrounding the alumina crucible core and by the emissivity of the alumina crucible. The Zircar block surrounds the heater and its very low thermal conductivity enables heat to be stored within the crucible. The main thermal path for heat to leave the stage is by radiation through the crucible surface. As the emissivity of alumina is hard to determine, the property was used as a means of providing fine tuning in order for results to correlate with experimental data.

In the heating stage, the temperature of the crucible is measured remotely through a thermocouple mounted underneath the heater. In the finite element model, a node was located at the same position as the thermocouple to create...
Table 1. Material properties with references given in square brackets.

| Material name        | Location in heating stage | FE model sensitivity | Density (kg m\(^{-3}\)) | Thermal conductivity (W m\(^{-1}\) K\(^{-1}\)) at 0 °C, 100 °C, 500 °C, 1000 °C, 3000 °C | Specific heat capacity (J kg\(^{-1}\) K) at 0 °C, 100 °C, 500 °C, 1000 °C, 3000 °C | Emissivity |
|----------------------|---------------------------|----------------------|--------------------------|---------------------------------------------------------------------------------|---------------------------------------------------------------------------------|------------|
| Alumina              | Crucible and surrounding housing and probe | 0.37% | 3960 [7] | 38 [8] 11 [8] 7 [8] 6 [8] 5 | 718 [8] 907 [8] 1089 [8] | 1089 1089 | 0.4 |
| Zircar ZYZ-3\(^a\)   | Porous Zirconia-based insulator surrounding alumina | 8.17% | 480 [3] | 0.07 0.07 0.086 0.13 0.25 | 753.6 | (see footnote 3) | 0.3 |
| Aluminium alloy      | Stage housing | 0.002% 0.04% | 2713 [9] 17 620 [8] | 46 [8] 51 [8] 69 [8] 69 69 | 880 [8] 937 [8] 1021 [8] | 1021 [8] 1130 | 0.82 [8] |
| Stainless steel      | Heating coils | 0.05% | 7916.5 [8] | 15.579 | 460.5 | 0.35 [6] |
| AREMCO 569           | Heat shield and cover plate | 0.08% | 3960\(^b\) | 38 11 7 6 5 | 718 907 1089 1089 1089 | N/A |
| Copper               | Aluminium-based ceramic adhesive used to pot the heater wire | N/A | 8800 [8] | 180 [8] | 379 [8] 397 [8] 419 [8] | 430 [8] 430 | 0.03 [11] |
| Tungsten             | Probe tip | N/A | 18 820 [8] | 0.12 [8] | 133 [8] 135 [8] 140 [8] | 143 [8] 143 | 0.38 |

\(^a\) Thermal conductivity values are generated by creating a function from manufacturers data (see footnote 3) and calculating values for temperatures stated.

\(^b\) AREMCO Products Inc., PO Box 517, 707-B Executive Blvd., Valley Cottage, NY 10989 (http://www.aremco.com/a2.html).
a measurement location to predict thermocouple temperatures. A comparison between the temperatures at the crucible surface to the thermocouple during transient analysis is shown in figure 6. The result shows that, in at least analytical terms, the thermocouple output and the temperature of the crucible should be very similar across the operating temperature range.

A typical illustrative result showing heat distribution through a central cross section of the heating stage for a given power is shown in figure 7. This is for the maximum temperature during a 900 °C set-point experiment (see figure 5) after the 10 min hold.

6. FE modelling of the micromanipulator

The 1D manipulator actuates a probe which has a tungsten needle tip to provide manipulation of the sample. The maximum operating temperature of the manipulator is about 90 °C and to provide thermal protection the probe is required to limit heat transfer to the piezoelectric motors. The FE model was used to design the probe and prove its feasibility before implementation in the heating stage. The probe is modelled using beam elements of appropriate section sizes. In addition, an aluminium alloy disc was modelled inside the crucible to represent the sample. The probe is modelled assuming a worst-case scenario, whereby the probe tip is submerged into the sample and is in perfect contact with the crucible. This generates the maximum possible heat transfer into the probe. The main section of the probe was constructed using alumina ceramic thin-walled tube due to its structural stability over a large temperature range, low thermal conductivity and high melting point. In reality, zirconia would be a better material choice but it was not possible to procure an appropriate section size. The FE model was used to simulate a single length of alumina acting as the extension arm. As with the modelling of the heating stage, perfect conduction is assumed between the elements. This is achieved by sharing nodes between beam elements and does not require the specification of thermal couplings. The results for a 650 °C set-point temperature showed that the temperature of the piezoelectric was 121 °C and would be damaged. To improve the thermal protection, a cylindrical copper block and larger square section of alumina were incorporated as indicated in figure 2.

The cylindrical copper block has holes drilled into either end to accommodate both sections of alumina and coupling is assumed perfect. However, the square-section alumina is attached using a friction fit connection at each corner. This minimizes conduction and heat transfer is dominated by radiation. The connection is modelled by creating a small gap of 0.1 mm between the copper block and the alumina through which a near-zero conductance is specified. The parts are all sufficiently close that radiative heat transfer is representative. The FE model analysis showed that with this arrangement the piezoelectric would remain within safe operating temperatures across the operating range of the heating stage. A comparison between the FE model predicted temperatures and temperatures measured during experiment (for 650 °C set point) is shown in figure 8. The results show reasonable agreement and validate the analytical approach. The figure also shows the temperature distribution across the probe with a single length of alumina.

Discrepancies between the experimental values and the FE model are expected due to limitations in both methods. The FE model assumes perfect conditions which are not always represented in the heating stage experiments. Even when using the calibrated model continual changes in the environment and heating stage performance are not accounted for and can result in errors when using the model to predict temperatures for different heat loads. In addition during experiments up to six measurements were taken by hand using various digital meters at a single interval. These measurements took up to 20 s to record and introduced errors as values are continuously changing. The introduction of measuring equipment (e.g. thermocouples) adds additional sources of heat loss which result in the stage behaving differently to when a sample is being heated. The extents of these errors are difficult to quantify due to their variability. It is reasonable to assume that the differences in experimental temperatures and FE model temperatures can be largely accounted for by these errors. However, as a safety precaution to any unexpected
Design of a micromanipulation system for high temperature operation in an ESEM

Figure 7. Temperature distribution through the heating stage after 10 min hold for a set point of 900 °C (see figure 5(c)).

Figure 8. Comparison of FE and experimental results for the manipulator probe design for a 650 °C set-point temperature after 1 h of heating.

| Location                        | Probe With Copper Block Arrangement | Probe without Copper Block |
|---------------------------------|-------------------------------------|---------------------------|
|                                 | Experiment Temp (°C) | F.E. Temp. (°C) | F.E. Model Prediction (°C) |
| 1 – Probe Tip                   | 605                    | 605.4             | 605.3                     |
| 2 – 25% along Alumina Rod from tip | 123.4                 | 119.2             | 173.9                     |
| 3 – Copper Block End            | 51.3                   | 52.4              | 121                       |

Figure 7. Temperature distribution through the heating stage after 10 min hold for a set point of 900 °C (see figure 5(c)).

In addition to protecting the piezoelectric transducers, the probe must not act as a heat sink and remove significant quantities of heat from the crucible. This could lead to localized cold regions being created in the sample. The FE model was used to check this and figure 9 shows the temperature distribution across the sample when the probe is attached. The result shows that no localized cold regions are formed in the sample.

The temperature distribution is shown extremely clearly across the crucible surface. The coils of the heating wires are closest at the centre and therefore peak temperatures in the crucible are developed in this region. However, the cooling water runs along one side of the heating stage and this creates a cooler region along one side. The effect on the sample is shifting of the hot region slightly away from the centre and towards the hotter side of the heating stage. It is important to note that the maximum temperature variation across the crucible is only 3 °C indicating that most experiments can be conducted in any region of the crucible.

7. Experimental validation

Experiments were conducted whereby thermocouples were bonded to key points of the heating stage. These bonding locations included the crucible surface along the 1D probe.
Figure 9. Temperature distribution across the sample in the crucible for 650 °C set-point temperature.

Figure 10. Comparison between experiment and FE model results for different operating conditions.

In these experiments, the 1D manipulator probe tungsten tip was used to pin the thermocouple to the crucible surface, which was filled with AREMCO 569 alumina-based ceramic adhesive [9] to ensure good thermal conduction. It was found that without bonding the thermocouple and the probe tip to the crucible surface incorrect temperatures were measured as the thermocouple tended to lift from the crucible surface. In this case, the thermocouple would only measure radiative heat which is significantly lower than the actual surface temperature. A comparison of FE and temperatures recorded during experiments is shown in figure 10. Each set of result uses different ramp inputs and peak temperatures. The results show reasonable correlation between the FE and experimental data.

For the 650 °C set-point experiment there is approximately 45 °C between the experimental result and the prediction at the end of the run. This may be associated with the errors for thermocouple measurement discussed in section 6.

At 950 °C, the discrepancy in figure 10 between the 900 °C set-point temperature and the temperatures measured during experiment is larger. This has not been resolved and is under investigation. The 900 °C set-point temperature experiment
was performed after the heating stage had been used for over its normal lifespan. The extra use may have caused the Zircar and adhesive cement around the heating coil to break down resulting in heat leakage and potentially causing the stage’s thermocouple to measure temperatures, which are not representative of the crucible (i.e. the original calibration is not valid). In addition, the addition of thermocouples wires and ceramic adhesive to the crucible may cause a large temperature gradient across the heating coil. This is due to heat loss through the thermocouple wires and the additional thermal mass of the ceramic adhesive. However, there is evidence that the stage power circuit compensates for this.

8. Demonstration of manipulation

The principle of manipulating a sample in the heating stage was proved using a small sample of aluminium alloy and the 1D manipulator. Before the experiment, the 1D manipulator probe tip was positioned on top of the sample. The probe tip applied a force on the surface such that it would indent the surface when the sample became semi-solid. The sample was heated using a heating load input used in FE model calibration. This heat load was demonstrated in experiment to provide reasonable agreement between the temperature at the crucible and the controller display temperature. At a heater controller display temperature of 650 °C, the 1D manipulator was activated and the probe tip moved across the sample, creating a hole in the surface. The results of the deformation are shown in figure 11. The results clearly show the hole made by the probe tip in the sample and the deformed grains around the hole surface. The quality of the images obtained demonstrates that the presence of the probe does not degrade image quality. Further details are given in [12].

In a second demonstration, a group of copper particles has been spread across the base of the crucible and the micromanipulator driven through at 900 °C (figure 12). A further illustration of the capacity to image particle–particle contacts under manipulation is given in [13].

9. Concluding remarks

The aim here was to test the design of a system for high temperature micromanipulation in an environmental scanning electron microscope with finite element modelling and experimental validation. A one-dimensional probe was used as a simple, inexpensive model of a three-dimensional micromanipulation system. The predictions from finite element modelling give reasonable agreement with experimental results from thermocouples although there are discrepancies at the highest temperatures. These may be associated with the degradation of the insulation in the heating stage with prolonged use. The use of the one-dimensional manipulator at high temperature in the ESEM has been successfully demonstrated by making a crater in the surface of an aluminium sample and by pushing copper power particles across the base of the crucible.

Acknowledgments

The authors are grateful to Mr Andrew Smith for carrying out some of the experimental validation and to the Engineering
and Physical Sciences Research Council for financial support under grant GR/S97910/01. They would like to express their thanks to Mr Baden Flavill, Mr Tony Crawford and Mr Graham Clarke for technical support and to FEI and Mr Ralph Knowles for technical support and cooperation.

References

[1] Danilatos G D 1988 Adv. Electron Phys. 71 109–250
[2] Cocks A C F, Gill S P A and Pan J 1999 Adv. Appl. Mech. 36 81
[3] I-Deas Version 11, UGS, Norwich House Knoll Road, Camberley, Surrey GU15 3SY, UK (http://www.ugs.com/products/nx/ideas)
[4] MAYA TMG (http://www.mayaht.com/products/thermal)
[5] Harris N 2005 Modern Vacuum Practice 3rd edn (Crawley, UK: BOC Edwards) www.modernvacuumpractice.net
[6] I-Deas 9 TMG Course Guide—Unit 14, Conductance Modelling, EDS Structural Dynamics Research Corporation, 2000 Eastman Drive, Milford, OH, 45150 (513)576-2400
[7] I-Deas 9 TMG Course Guide—Unit 17, Conductance Modelling, EDS Structural Dynamics Research Corporation, 2000 Eastman Drive, Milford, OH, 45150 (513)576-2400
[8] Kaye G W and Laby T H 1995 Table of Physical and Chemical Constants 16th edn (Essex, UK: Longman)
[9] Matweb—Material Properties Website (www.matweb.com)
[10] I-Deas Materials Database, EDS Structural Dynamics Research Corporation, 2000 Eastman Drive, Milford, OH, 45150 (513)576-2400
[11] Yunus A and Cengel 1998 Heat Transfer: A Practical Approach (New York: McGraw-Hill) p 31
[12] Smith A J, Atkinson H V, Hainsworth S V, Dong H B and Haghaeyghi R 2006 Solid State Phenom. 116–117 700–3
[13] Smith A J, Atkinson H V, Hainsworth S V and Cocks A C F 2006 Scripta Materialia 55 707–10