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Research Paper

RAMAN ANALYSIS AND EXPERIMENTAL INVESTIGATION OF NICHROME AND ALUMINIUM NITRIDE MICROTUBULAR COIL HEATERS: A 2D APPROACH

G Sureshkannan[1]* and M Velliangiri[1]

Corresponding Author: G Sureshkannan, ✉ *sureshkannan_g@rediffmail.com*

Raman spectroscopy is a spectroscopic technique based on inelastic scattering of monochromatic light, usually from a laser source. Surface-Enhanced Raman Scattering (SERS) combines modern laser spectroscopy with the exciting optical properties of metallic structures, resulting in strongly increased Raman signals when molecules are attached to micrometer-sized structures. Micro tubular coil heaters have been widely investigated because of their extensive applications in Microsystems. These are presently manufactured by using Nichrome, a commercial alloy suitable for all types of macro/micro tubular heaters, but can be used for relatively low temperature applications. This paper aims in determining a micro tubular coil heater material suitable for high temperature applications. The material characterization was performed using Raman spectrometer and the geometric optimization for the micro tubular coil heater was performed by using COMSOL Multiphysics software. The characteristic dimensions of the micro tubular coil heater are varied and the results are discussed and compared to each other. The result of temperature distribution of 2-Dimensional thermal analysis is applied to thermal stress analysis, enabling analysis of the time dependent thermal stress of Nichrome and Aluminium Nitride micro tubular coil heaters.

Keywords: Raman spectroscopy, Micro tubular coil heaters, Thermal stress, Temperature distribution, Rupture point, Comsol analysis

INTRODUCTION

Raman spectroscopy is a spectroscopic technique based on inelastic scattering of monochromatic light, usually from a laser source. Inelastic scattering means that the frequency of photons in monochromatic light changes upon interaction with a sample. Photons of the laser light are absorbed by the

[1] Coimbatore Institute of Technology, Coimbatore 641014, India.

sample and then reemitted. Frequency of the reemitted photons is shifted up or down in comparison with original monochromatic frequency, which is called the Raman Effect. This shift provides information about vibrational, rotational and other low frequency transitions in molecules. Raman spectroscopy can be used to study solid, liquid and gaseous samples. A Raman microscope consists of a standard optical microscope, an excitation laser, a monochromator and a sensitive detector. The correct selection of the laser wavelength is an important consideration for Raman spectroscopy. With modern Raman instruments, often several laser wavelengths may be employed to obtain the best detection limit of the Raman signal with sensitivity. For example, many samples, especially organic or biological, will be quite fluorescent in nature. Exciting these samples with a laser in the green (532 nm) light may promote this fluorescence, and this fluorescence background may suppress any underlying Raman spectrum to such an extent that it is no longer detectable. In this case, the use of a laser in the red (633 nm) or near infrared (NIR) (785 nm) region may provide a solution. With the lower photon energy, a red or NIR laser may not promote the electronic transition and so the Raman scatter may be far easier to detect. The cross-section for fluorescence is 10-16 cm^2 per molecule, but for Raman scattering, the value is between 10-31 cm^2 per molecule (Jimenez-Sandoval, 2000; Thorley *et al.*, 2006; Misra *et al.*, 2007; Gaft and Nagi, 2008; and Alison and Bernhard, 2009).

There has been a little work on the Raman spectroscopy studies on nitride based alloys. Because Raman scattering of materials with metallic properties is restricted by prohibitive selection rules, low penetration depth of the incident light, etc.

COMSOL Multiphysics is a finite element analysis and solver software package for various physics and engineering applications, especially coupled phenomena, or multiphysics. It is suitable software package for small scale electro thermal analysis since a separate MEMS module is present in it. COMSOL provides better solution accuracy and consistency. COMSOL has a separate MEMS module which represents coupled processes in microelectromechanical and microfluidic devices.

MICRO TUBULAR COIL HEATERS OVERVIEW

The first micro tubular heaters have been designed for plastics injection molding industry where hot runner system is used and represent the best solution to heat nozzles with very small diameters requiring specific power and very high temperatures. Micro tubular coil heaters are made as rectangular, square and circular sections, with or without built-in thermocouple. Micro tubular heaters are swaged and compacted with magnesium oxide and a helical or straight resistance element. The main features of these heaters are 3600 heated area, readily confirms to surface, fast response and quick heat transfer, helical coil design for superior performance, good resistance to corrosion and it can easily be coupled with J or K type thermocouple. It also provides the best combination of physical strength, high emissivity and good thermal conductivity to heat hot runner bushing/nozzles mainly for multi cavity hot runner PET perform moulds and thin wall container moulds.

CHARACTERIZATION AND EXPERIMENTAL INVESTIGATION

Identification of Candidate Material

Presently the micro tubular coil heater wires are manufactured using Nickel-Chrome alloys, which are resistant to high temperatures. 80/20 Nickel-Chrome alloys containing long life additions which make it eminently suitable for applications such as nichrome resistance wire, subject to, frequent switching and wide temperature fluctuations. Nicrome80:20 is a non-magnetic alloy which consists of 79% nickel, 19% chromium, 0.8% Iron, 0.5% Silicon, 0.4% Carbon and 0.3% Manganese by weight, and is widely used in heating elements because of its relatively high resistivity. A relatively low temperature coefficient of resistance with a high resistivity makes it suitable for control resistors. Nichrome wire has two key virtues, firstly it is almost 100% efficient in converting electrical energy into heat, and secondly provided the temperature is not too high it will not oxidize in air (Table 1).

Table 1 Physical Properties of Nichrome and AlN

Material/Properties	Nichrome	AlN
Melting Point, ºC	1400	2200
Resistance at room temperature, ohm m	1.0×10^{-6}	1.3×10^{-6}
Thermal Conductivity, W/m ºC	11.3	120
Density, kg/m^3	8400	3260
Modulus of elasticity, N/m2	2.2×1011	3.31×10^{11}
Thermal Expansion Coefficient, K^{-1}	13.4×10^{-6}	5.6×10^{-6}
Operating temperature, ºC (As micro tubular heating element)	750	1450

The disadvantage of micro tubular coil heater and all types of heaters is the change of resistance with the change in dimensions. The resistance increases with the increase in length and it decreases with increase in cross sectional area, i.e., diameter. Table 2 shows the increase in resistance with its temperature of the nichrome heater wire.

Table 2: Change of Resistance with Temperature of Nichrome Heater Wire

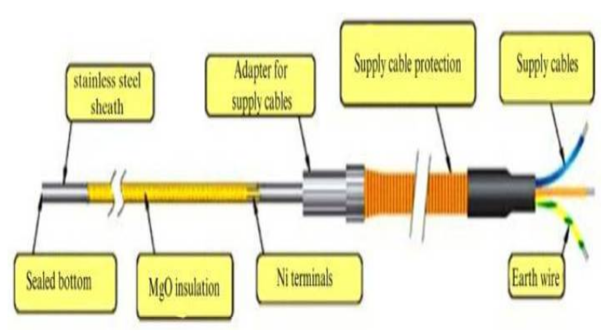
°C	20	95	208	316	427	537	689
% Increase in Resistance	0	0.8	2.1	3.4	4.7	6.3	5.8

It is evident from the table that the resistance of the heater material increases with its temperature. Because of the increase in resistivity with the reduction in cross sectional area, its operating temperature is limited to 750 °C, which is very low for most of the heating applications.

In order to use micro tubular coil heaters more than 750 °C, we select Aluminium Nitride (AlN) as the material for heating element for which the melting point is 2200 °C, which is almost 1.5 times the melting point of Nichrome. An attempt was already made with Titanium Nitride for micro tubular coil heater by us (Sureshkannan *et al.*, 2012). Titanium Nitride has already been tried for heaters and micro heaters (de Moor *et al.*, 1999; Creemer *et al.*, 2008). The higher melting point of the AlN material made us to select it as a candidate material for high heat applications even though its resistivity is relatively low. Even though AlN is tested for high temperature applications (Kazan *et al.*, 2006; Trodahl *et al.*, 2006; Xiaojun *et al.*, 2006; and Oliveira *et al.*, 2001), it is not tried for micro tubular coil heaters.

Aluminum nitride has a hexagonal crystal structure and is a covalent bonded material. The material is stable to very high temperatures in inert atmospheres. A layer of aluminum oxide forms which protects the material up to 1370 °C. Above this temperature bulk oxidation occurs. Aluminum nitride is stable in hydrogen and carbon dioxide atmospheres up to 980 °C.

Figure 1: Components of Micro Tubular Coil Heater



CHARACTERIZATION OF MATERIALS

The Raman spectra of two materials are obtained using 785nm Raman spectrometer and these are shown in Figures 2 (Nichrome) and 3 (AIN). From the literature and these two spectra, it is evident that the AIN material has very high melting point and low thermal Expansion Coefficient.

The ratio of the intensity of the Raman peaks in Figure 2 sharpens at higher temperatures. This shows that the crystal structure of Nichrome gets changed at high temperature ranges which makes it is not suitable for high temperature applications of microtubular heaters. The ratio of the intensity of the Raman peaks in Figure 3 changes little over the entire temperature range. This shows that the crystal structure of AIN remained stable at all the temperature ranges (Xiaojun *et al.*, 2006) which makes it suitable for high temperature ranges and can be used for high temperature microtubular heater applications. Also it has high modulus of elasticity which makes the material suitable for heavy load.

Figure 2: Raman Spectra of Nichrome at Different Temperatures

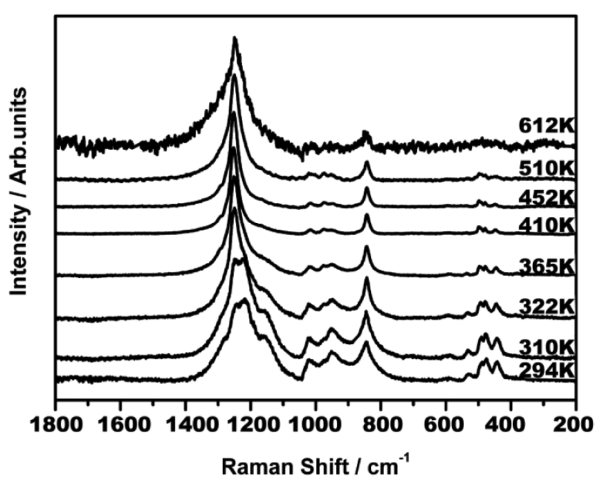
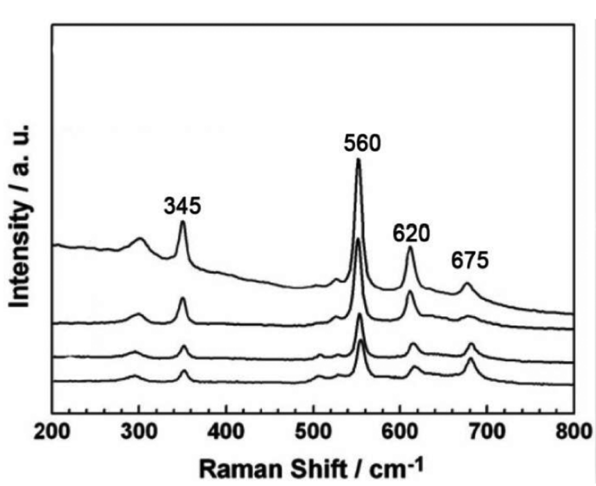


Figure 3: Raman Spectra of AlN at Different Temperatures



EXPERIMENTAL

We made a micro tubular coil heater with AIN as heating element without changing the sheath material and insulation compound. The external sheath is made in CrNi steel in which inside a AIN resistive wire, uniformly distributed, is insulated in a high compressed MgO compound The important parameters considered for manufacturing micro tubular coil heaters are resistant wire material, sheath material, insulation material, maximum sheath temperature, and type of thermocouple. The AIN micro tubular coil heater is made in four different sizes as like Nichrome micro tubular coil heater and tests were carried out. The test system consists of a DC power source, voltmeter, ammeter, variable resistor, micro tubular coil heater and J-type thermocouple. The power is applied by varying the current and voltage and the tests were carried out until the heater elements failed. The temperature and resistance for both the micro tubular coil heaters, at the point of rupture were obtained and the change of temperature and resistance with its diameter is calculated and shown in Table 3.

Table 3: Rupture Temperature of Nichrome and AlN

Size (Diameter of the Coil Wire Element) in mm	Rupture Temperature in K	
	Nichrome	AlN
0.5	751.13	1729.59
0.25	702.18	1687.06
0.125	665.29	1498.24
0.075	602.08	1310.05

SIMULATION OF TEMPERATURE DISTRIBUTION OF MICRO TUBULAR HEATERS

The geometric optimization for the micro tubular heaters has been performed by simulating various geometries using COMSOL multiphysics. It is suitable software package for small scale electro thermal analysis since a separate MEMS module is present in it Velmathi *et al.* (2010). The Electro-Thermal module of the COMSOL multiphysics tool has been used for simulation to achieve the desired function of the micro tubular heater. The Nichrome and AlN micro tubular coil heaters are modeled in four different sizes and simulation tests were carried out. The

Figure 4: Temperature Distribution of Nichrome Micro Tubular Coil Heater (Diameter 0.5 mm)

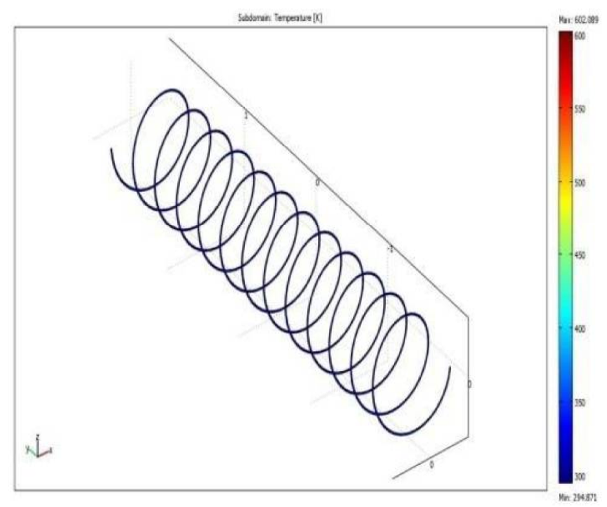
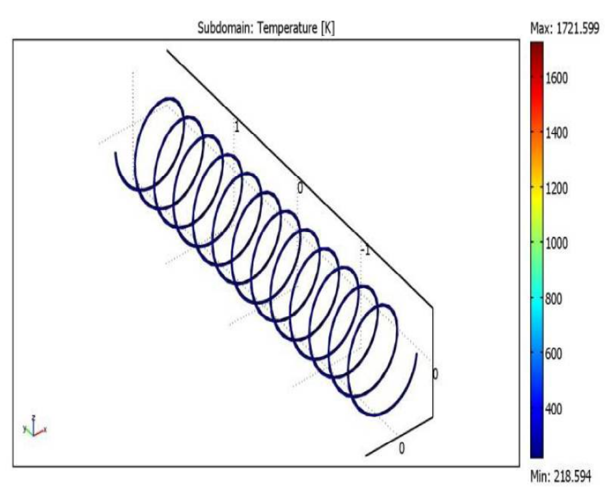


Figure 5: Temperature Distribution of AlN Micro Tubular Coil Heater (Diameter 0.5 mm)



temperature of the micro tubular coil heaters for various input parameters (current, voltage and wattage), including the point of rupture were obtained and compared with the experimental results.

SIMULATION OF THERMAL STRESS AND DEFLECTION OF MICRO TUBULAR HEATERS

Thermal stress is an important parameter where heating is employed. The result of temperature distribution of 2-Dimensional thermal analysis is applied to thermal stress analysis, enabling analysis of the time dependent thermal stress of Nichrome and Aluminium Nitride micro tubular coil heaters. Figures 12 and 13 show the VonMises stress induced in Nichrome and Aluminium Nitride.

RESULTS AND DISCUSSION

It is evident from the Table 2 that the resistance of the heater material increases with its temperature. Because of the increase in resistivity with the reduction in cross sectional area, the operating temperature of Nichrome micro tubular coil element is limited to 602.89 K, which is very low for most of the heating applications. But the operating temperature of AIN micro tubular heating element is 1729.59 K for the diameter of 0.5 mm. The Table 3 shows the rupture temperature of both Nichrome and AIN micro tubular heating coils at various cross sections.

From the Table 3 the rupture temperature of both micro tubular coil heaters decreased with decrease in its diameter since the resistance of the heater material increases with its decrease in diameter.

From the Table 2, it is proved that the resistance of the heater material increases with its temperature and the resistivity of the material increases with decrease in diameter. And also from the Table 3, the maximum rupture temperature of the 0.075 mm Nichrome heater is 602.089 K and AIN heater is 1310.05. The experimental and simulated results for all the dimensions are presented in the Figures 6-11, including the maximum operating temperatures. The rupture temperature of AIN micro tubular heater is 2.17 times more than that of Nichrome. This shows that AIN micro tubular heaters can be used for high temperature applications. Due to low resistivity of the AIN, the power applied to AIN heater is higher than that of Nichrome heater for the same dimension. But due to the higher melting point and high operating temperature of AIN, it is suitable for high temperature applications which made the AIN a suitable candidate for micro tubular coil heaters.

Figure 6: Current vs. Temperature of 0.075 mm Diameter Nichrome Wire Element

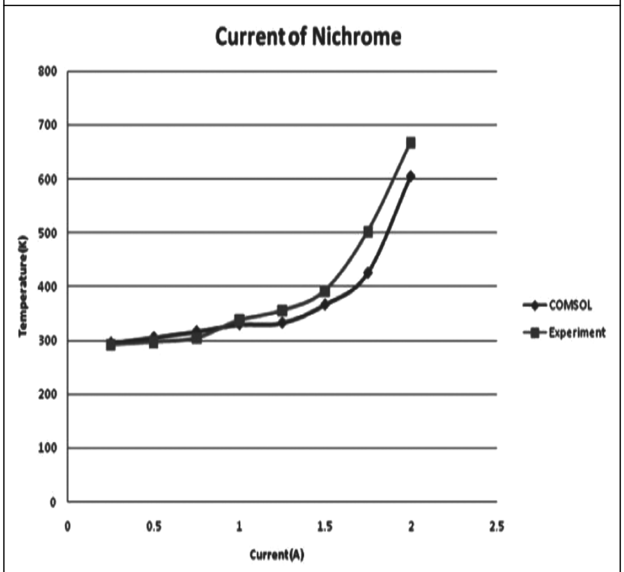


Figure 7: Current vs. Temperature of 0.075 mm Diameter AlN Wire Element

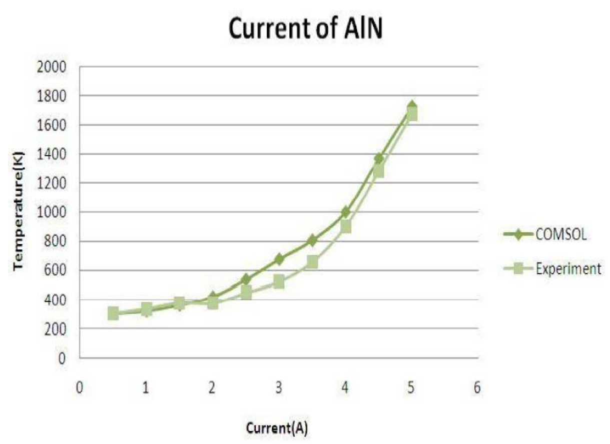


Figure 8: Voltage vs. Temperature of 0.075 mm Diameter Nichrome Wire Element

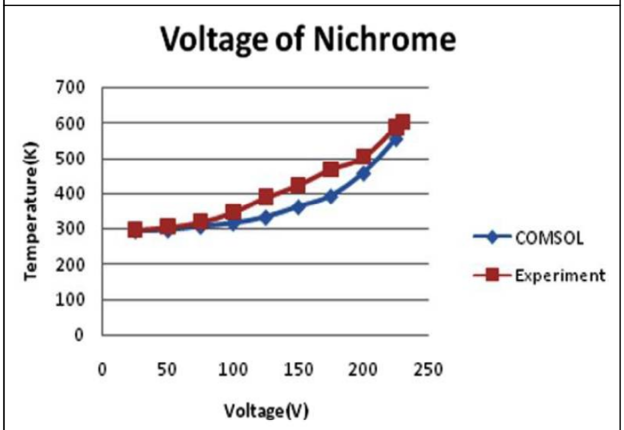


Figure 9: Voltage vs. Temperature of 0.075 mm Diameter AlN Wire Element

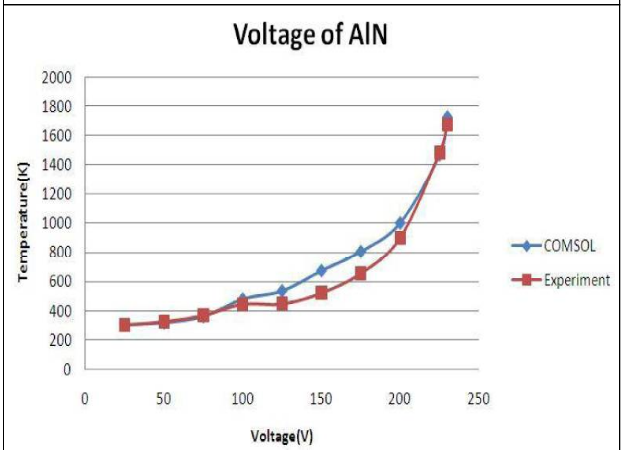


Figure 10: Wattage vs. Temperature of 0.075 mm Diameter Nichrome Wire Element

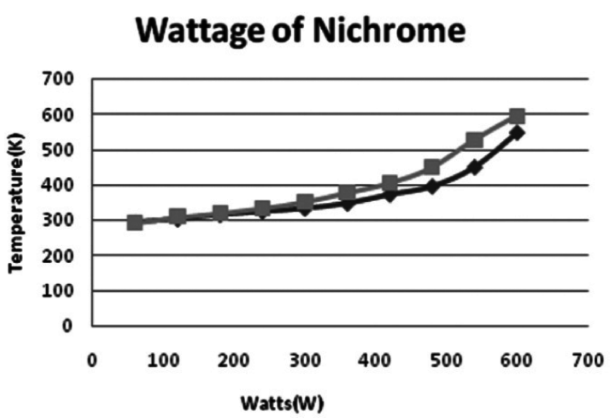
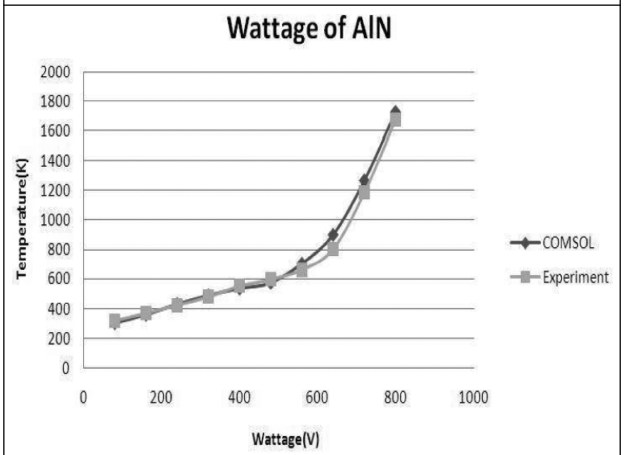


Figure 11: Wattage vs. Temperature of 0.075 mm Diameter AlN Wire Element



The temperature distribution in a part may cause thermal stress effects i.e., stress caused by thermal expansion or contraction of the material. Thermal stress effects were simulated by coupling a steady state heat transfer analysis and a structural analysis. First a heat transfer analysis was performed to determine the temperature distribution and these temperature results were applied as load in a structural analysis to determine the stress and displacement caused by the temperature loads. Figures 12 and 13 show the thermal stress analysis of Nichrome and

AlN. From the Figures 12 and 13, it was observed that the stress caused due to the thermal load is 4.53×104 N/m^2 for Nichrome micro tubular heater and is 4.498×104 N/m^2 for AlN micro tubular heater. This shows that the thermal stress is comparatively low for AlN micro tubular coil heater.

Figure 12: Thermal Stress Analysis of Nichrome

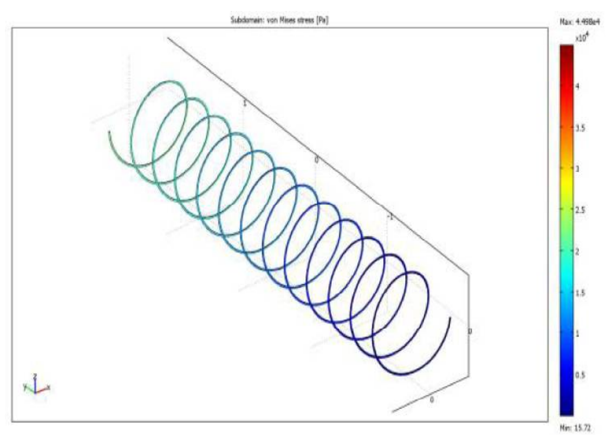
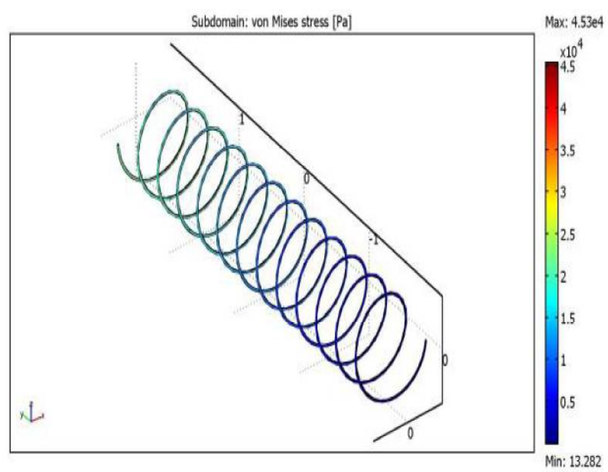


Figure 13: Thermal Stress Analysis of AlN



Figures 14 and 15 are the result of structural analysis with the temperature load as input (Vanni and David, 2006) and these give the mechanical deformation of Nichrome and AlN micro coil heaters for a particular thermal load. From these two figures it is found that the deformation caused by temperature load is comparatively less for the AlN micro tubular coil heater.

Figure 14: Mechanical Deformation of Nichrome

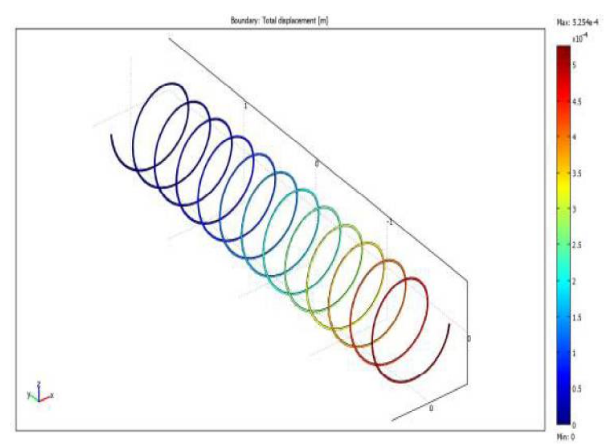
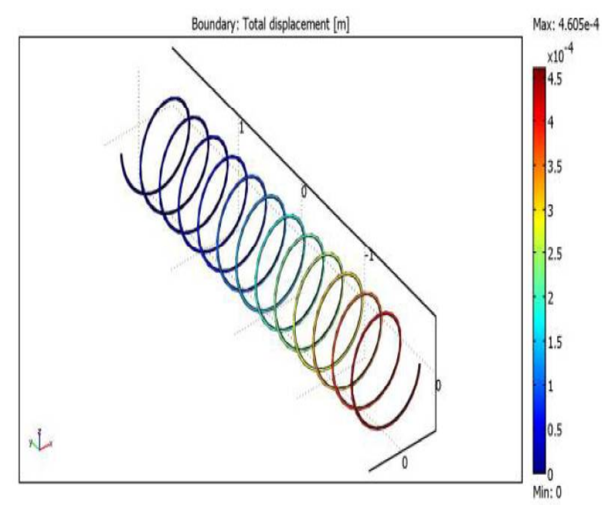


Figure 15: Mechanical Deformation of AlN



From the Raman spectra of these two materials, it is observed that the structure of AlN is stable even at high temperatures (Xiaojun *et al.*, 2006) and from the Comsol analysis, it is found that the thermal stress and mechanical deformation are comparatively less in AlN micro tubular heater which made AlN a suitable candidate material for high temperature applications.

CONCLUSION

This paper describes the characterization, experimental investigation and analysis of Aluminium Nitride (AlN) micro tubular coil heater using Raman spectroscopy and Comsol multiphysics. The material characterization was performed using Raman spectrometer and the geometric optimization for the micro tubular coil heater was performed by simulating a wide range of possible geometries using COMSOL multiphysics, a commercial Finite Element Analysis (FEA) package. The characteristic dimensions of the micro tubular coil heater structures are varied and the results are discussed and compared to each other. The temperature distribution of result of 2-dimensional thermal analysis is applied to thermal stress analysis Nichrome and Aluminium Nitride micro tubular coil heaters and Aluminium Nitride coil heaters provided satisfactory results over Nichrome coil heaters.

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