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# CHARACTERIZATION AND EXPERIMENTAL INVESTIGATION OF TiN MICRO TUBULAR COIL HEATER USING RAMAN SPECTROSCOPY

# CHARAKTERYSTYKA I BADANIA DOŚWIADCZALNE MIKRO-GRZAŁEK Z TiN METODĄ SPEKTROSKOPII RAMANOWSKIEJ

Raman spectroscopy is a commonly used tool in bio-diagnostics and micro sensor technology. Surface-enhanced Raman scattering provides high signal enhancements especially at micro and nanostructured metallic surfaces. Micro tubular coil heaters have been widely investigated because of their extensive applications in PET Preformed Moulds, Hot Runner Nozzles & Bushings and Thin Walled Container Moulds and other Microsystems. This paper describes the characterization, experimental investigation and analysis of Titanium Nitride (TiN) micro tubular coil heater using Raman spectroscopy and Comsol multi-physics. 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 COMSOLTM, a commercial Finite Element Analysis (FEA) package. The characteristic dimensions of the microstructures are varied and the results are discussed and compared to each other. The simulated results of micro tubular heaters having an improved temperature distribution over the sensing area and a higher density of integration is presented in this paper.

*Keywords*: Micro tubular coil heaters, Raman Spectroscopy, Thermal profile

Spektroskopia ramanowska jest powszechnie stosowanym narzędziem w bio-diagnostyce i technologii mikroczujnika. Powierzchniowo wzmocniona spektroskopia ramanowska zapewnia wysokie wzmocnienie sygnału zwłaszcza na mikro i nanostrukturalnych powierzchniach metalowych. Mikro-grzałki (mikro cewki) były szeroko badane ze względu na ich szerokie zastosowanie w formowaniu wtryskowym, podgrzewaczach dysz i tulej, i cienkościennych formach i innych mikrosystemach. Artykuł opisuje charakterystykę, badania eksperymentalne i analizę mikro-grzałki rurowej z azotku tytanu (TiN) za pomocą spektroskopii ramanowskiej i oprogramowania COMSOL Multiphysics. Charakterystykę materiału przeprowadzono za pomocą spektrometru Ramana a geometryczną optymalizację dla mikro-grzałki prowadzonosymulując szeroki zakres możliwych geometrii, za pomocą COMSOLTU, komercyjnego pakietu do analizy metodą elementów skończonych. Charakterystyczne wymiary mikrostruktur są zróżnicowane, a wyniki zostały omówione i porównane ze sobą. Symulowane wyniki mikro-cylindrycznych grzejników o ulepszonym rozkładzie temperatur w obszarze wykrywania i większej gęstości integracji zostały przedstawione w niniejszym artykule.

## 1. Introduction

*Raman spectroscopy and COMSOL Multiphysics overview*

Raman spectroscopy is an optical chemical analysis technique that measures the intensity of inelastically scattered light over a range of frequencies [1]. During the Raman scattering process, a small portion of the incident photons (1 in $10^6$-$10^7$) are scattered inelastically due to their interaction with the vibrations or rotations of atoms or molecules. The inelastic exchange occurs because the photons either lose or gain energy from the material under examination. Photons of the laser light are absorbed by the 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 [2-5]. A Raman system typically consists of four major components: Excitation source (Laser), Sample illumination system and light collection optics, Wavelength selector (Filter or Spectrophotometer) and Detector (Photodiode array, CCD or PMT). The performance of Raman spectrometer depends on the wave length of laser. And the intensity of the Raman spectra is inversely proportional to the fourth power of wave length of the laser. When light from a laser source , with frequency $\omega_i$, incidents on a sample which has frequency $\omega_{o,}$ due to the phono-phonon interaction, the source laser scattered from the sample $\omega_{s,}$ and the output frequency will be $\omega_s = \omega_i \pm \omega_o$. These components $\omega_i + \omega_o \omega_i$-$\omega_o$ are called as anti-stokes and stokes Raman scattering respectively.

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Resolution can be increased either by changing the wavelength of the laser or by cleaving the sample or by using a confocal Raman instrument. Raman spectrometer can be used for identifying stress and temperature dependence of the materials [6-10]. COMSOL Multiphysics is suitable software package for small scale electro thermal analysis since a separate MEMS module is present in it [11, 12]. COMSOL provides better solution accuracy and consistency. COMSOL brings unsurpassed multi-physics capability simply not found in other codes. COMSOL has a separate MEMS module which represents coupled processes in microelectromechanical and microfluidic devices. It incorporates specific multiphysics couplings for applications such as film damping, piezoelectricity and fluid-structure interaction.

*Micro tubular coil heaters overview*

Micro tubular coil heaters are adapted to a variety of applications. This is because it can be formed into any shape. These heaters have been widely used in PET preformed moulds, injection moulding machine nozzle, hot runner nozzles & bushings, thin walled container moulds, packaging machineries and other micro systems.

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 360° 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 & good thermal conductivity to heat hot runner bushing / nozzles mainly for multi cavity hot runner PET perform moulds & thin wall container moulds.

## 2. Materials and experimental

*Candidate material identification*

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 (burn out) in air.

TABLE 1
Physical Properties of Nichrome

| Material / Properties | Nichrome | TiN |
|---|---|---|
| Melting Point | 1400°C | 2930°C |
| Resistance at room temperature | $1.0\times10^{-6}$ ohm m | $1.3\times10^{-6}$ohm m |
| Thermal Conductivity | 11.3 W/m°C | 19.2 W/m°C |
| Density | 8400 kg/m$^3$ | 5430 kg/m$^3$ |
| Modulus of elasticity | $2.2\times10^{11}$ N/m$^2$ | $2.51\times10^{11}$ N/m$^2$ |
| Thermal Expansion Coefficient | $13.4\times10^{-6}$ K$^{-1}$ | $9.35\times10^{-6}$ K$^{-1}$ |
| Operating temperature (As micro tubular heating element) | 750°C | 1900°C |

The main disadvantage of nichrome wire is that it cannot be soft soldered due to the operating temperature and must therefore be crimped or silver soldered. The another 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

| $^0$C | 20 | 93 | 204 | 315 | 427 | 538 | 649 |
|---|---|---|---|---|---|---|---|
| % Increase in resistance | 0 | 0.8 | 2.0 | 3.3 | 4.8 | 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 900°C, which is very low for most of the heating applications.

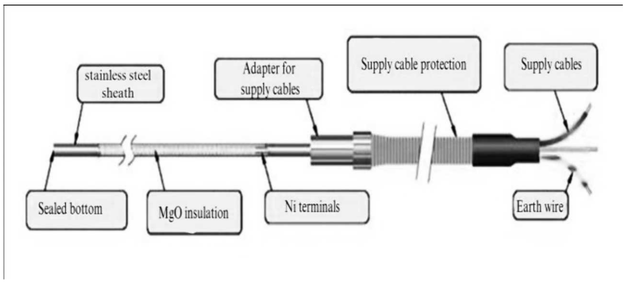


Fig. 1. Components of micro tubular coil heater

In order to use micro tubular coil heaters more than 900°C, we select Titanium Nitride (TiN) as the material for heating element for which the melting point is 2930°C, which is almost double the melting point of Nichrome. The higher melting point of the TiN material made us to select it as a candidate material for high heat applications even though its resistivity is relatively low. Even though TiN is used as a heater material for MEMS hotplates [13, 14], it is not used as heater material for micro tubular coil heaters.

## 3. Raman characterization of materials

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

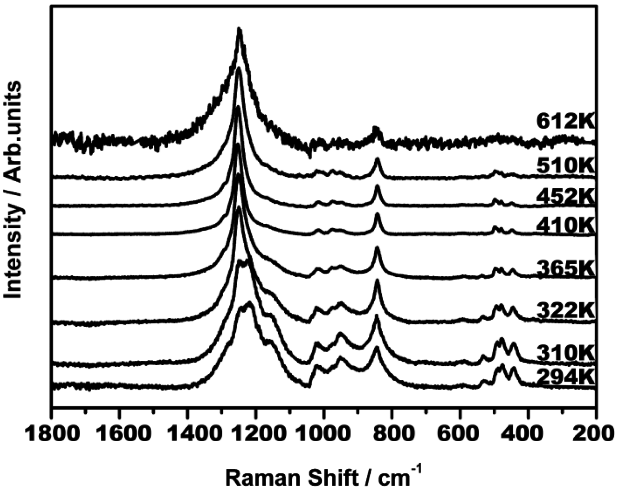


Fig. 2. Raman Spectra of Nichrome at different temperatures

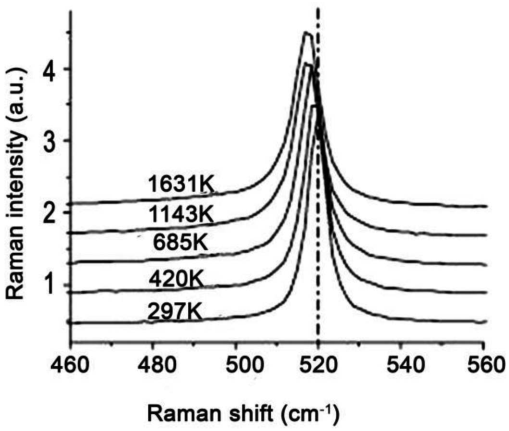


Fig. 3. Raman Spectra of Nichrome at different temperatures

The ratio of the intensity of the Raman peaks in Fig. 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 Fig. 3 changes little over the entire temperature range. This shows that the crystal structure of TiN remained stable at all the temperature ranges [15] 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.

## 4. Experimental

We made a micro tubular coil heater with TiN as heating element without changing the sheath material and insulation compound. The external sheath is made in CrNi steel in which inside a TiN 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 TiN 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 figure.

## 5. Simulation of the thermal behaviour of the micro tubular heater

The geometric optimization for the micro tubular heaters has been performed by simulating various geometries using COMSOL multiphysics. The Electro-Thermal module of the

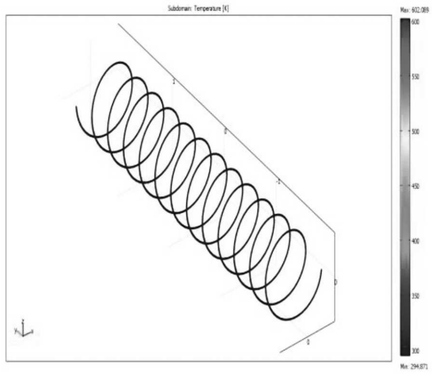

Fig. 4. Temperature distribution of Nichrome micro tubular heater coil

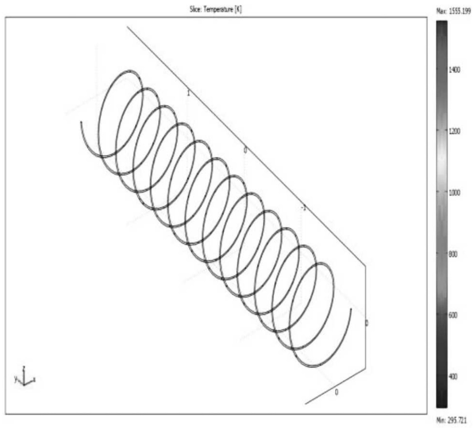

Fig. 5. Temperature distribution of TiN micro tubular heater coil

COMSOL multiphysics tool has been used for simulation to achieve the desired function of the micro tubular heater. The Nichrome and TiN micro tubular coil heaters are modelled in four different sizes and simulation tests were carried out. The 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.

## 6. Results & 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 TiN micro tubular heating element is 1555.19 K for the least cross section. The Table 3 shows the rupture temperature of both Nichrome and TiN micro tubular heating coils at various cross sections.

TABLE 3

Rupture temperature of Nichrome and TiN

| Size (Diameter of the coil wire element) in mm | Rupture temperature in K | |
|---|---|---|
| | Nichrome | TiN |
| 0.5 | 803.16 | 1868.06 |
| 0.25 | 725.08 | 1785.07 |
| 0.125 | 662.19 | 1692.30 |
| 0.075 | 602.89 | 1555.19 |

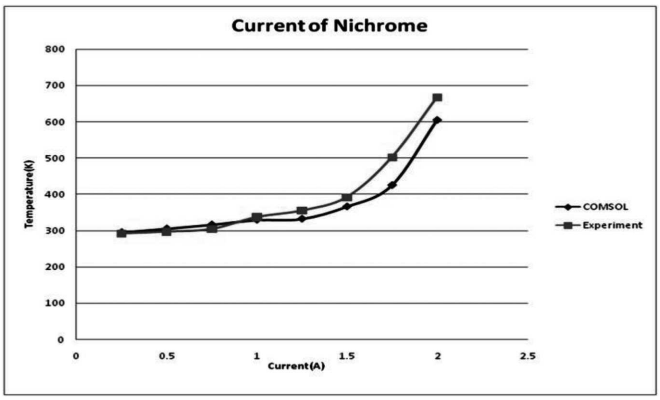


Fig. 6. Current Vs Temperature of 0.075 mm diameter Nichrome wire element

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.

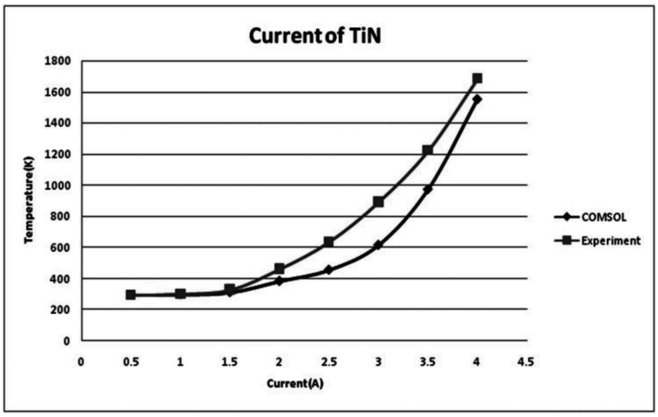


Fig. 7. Current Vs Temperature of 0.075 mm diameter TiN wire element

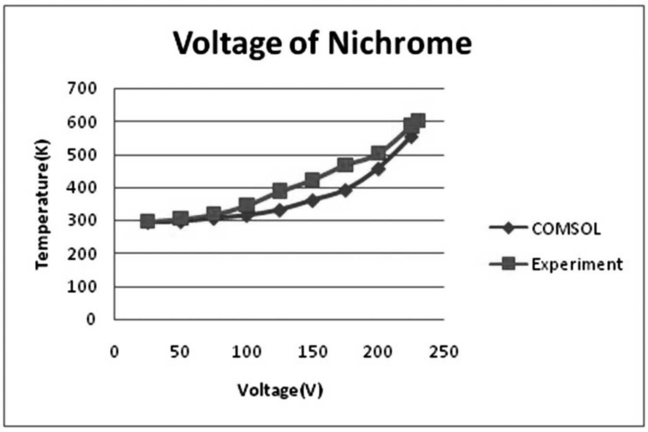


Fig. 8. Voltage Vs Temperature of 0.075 mm diameter Nichrome wire element

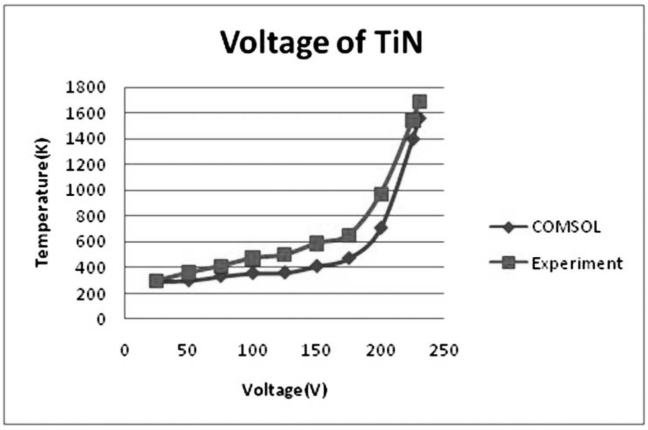


Fig. 9. Voltage Vs Temperature of 0.075 mm diameter TiN wire element

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 602.89 K and TiN heater is 1555.19 K. 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 TiN micro tubular heater is 2.58 times more than that of Nichrome. This shows that TiN micro tubular heaters can be used for high temperature applications. Due to low resistivity of the TiN, the power applied to TiN heater is higher than that of Nichrome heater for the same dimension. But due to the higher melting point and high operating temperature of TiN, it is suitable for high temperature applications which made the TiN a suitable candidate for micro tubular coil heaters.

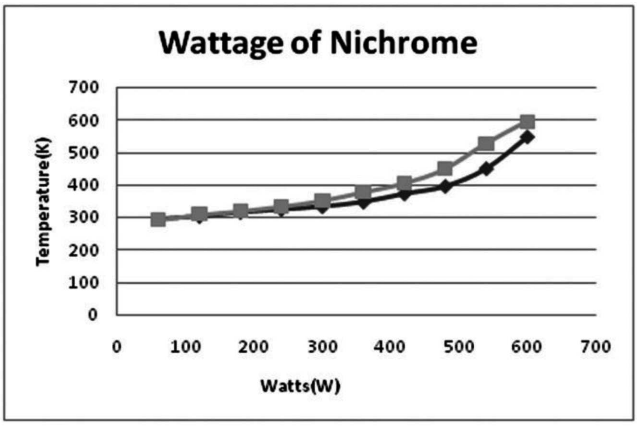


Fig. 10. Wattage Vs Temperature of 0.075 mm diameter Nichrome wire element

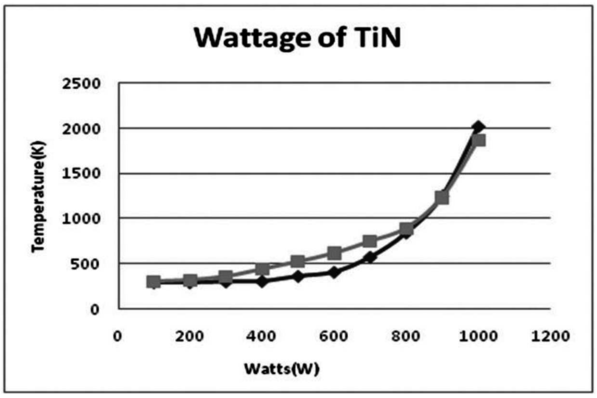


Fig. 11. Wattage Vs Temperature of 0.075 mm diameter TiN wire element

## 7. Conclusion

This paper describes the characterization, experimental investigation and analysis of Titanium Nitride (TiN) 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.

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