# Effect of Annealing Temperature on Sol-gel Grown TiC Nano Particles

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## Abstract

*We have synthesized TiC powders using sol-gel process at temperatures 1100 $^0$C and above, which resulted in cubic TiC phases with 4.330Å lattice parameter. Fine nano powders were obtained at 1100°C. The effect of annealing conditions in the temperature range 1100 - 1500°C resulted in grain broadening with increasing temperature. The repetitive cyclic voltammetry of TiC revealed good electrode like electrochemical properties. Temperature dependent electrical conduction exhibited insulating behaviour up to 600 $^o$C while semiconducting behaviour was observed at high temperatures.*

## 1. Introduction

Titanium carbide (TiC) is one of the most important compounds among the refractory transition metal carbides due to its promising physical and chemical properties [1]. TiC has been successfully integrated into various structural components like grinding and cutting tools, wear-resistant coatings, magnetic recording heads etc. TiC powders are usually synthesized via the reaction of titanium dioxide and carbon in the temperature range of 1700 – 2300 °C for 10 - 24 h [1]. Recently, carbothermal reduction [2], two stage refluxing method [3], co-reduction in an autoclave [4] *etc.* have also been used to synthesize TiC. Sol–gel process is a well-known chemical route to prepare oxide-based materials. Since solution based processing occurs at molecular level, fine-scale mixing of reactants is expected to lower the crystallization- temperature and restricts the crystallite growth. Moreover, the use of molecular precursors and the control of the synthesis conditions make it possible to prepare homogeneous and pure multi component systems. Sol gel with microwave carbothermal reduction has been used to prepare TiC powders [5]. In this work we have prepared TiC nano particles using sol-gel method and the effect of annealling temperature and/or time is shown to produce different crystallite sizes in TiC.

## 2. Experimental Details

TiC was synthesized using saccharose ($C_{12}H_{22}O_{11}$) as a carbon source. Saccharose was first dissolved in acetic acid ($CH_3COOH$). Titanium iso-propoxide ($Ti[OCH(CH_3)_2]_4$) was then added till the gel formation took place. Continuous stirring was done to evaporate the excess acetic acid. A thick gel thus obtained was dried at ~100 $^o$C. Based on the thermo-gravimetric analysis, the dried gels were calcined at 950, 1100, 1350, and 1500$^o$C for an interval of 5-18 h in argon atmosphere. For identification of phase(s) and morphology, an X-ray diffractometer (Thermoelectron model ARL X/TRA) was used. For conductivity measurements, a LCZ meter (model HP 4192A) was employed in the temperature range 30-900$^o$C. Cyclic voltammeter (CV) tests were performed using Metrohm Autolab electrochemical workstation.

## 3. Results and Discussion

### 3.1 Structural Characterization

The thermal stability and the oxidation resistance of the as-prepared TiC powders were investigated by thermo gravimetric analysis in the temperature range 50- 900°C. It was observed that TiC oxidation begins about 200°C followed by a rapid weight loss up to 750 $^o$C and it remains stable above 800 $^o$C at a value of ~ 18 % of initial value. Accordingly, the dried powders were calcined in the temperature range ~ 950 - 1500$^o$C. X-ray diffraction (XRD) patterns of TiC powders are shown in Fig.1. The 950 $^o$C treated sample (dotted curve) shows some additional phases of $Ti_2O_3$, $Ti_4O_7$, $TiO_2$ etc. as reported elsewhere [6]. An increase in temperature to 1100 $^o$C produced cubic *Fm*3*m* type TiC powders and no secondary phases were detected. The inset shows the relative broadening of (111) peak in the powders treated at 1100, 1350 and 1500 $^o$C. Scherrer's formula was used to estimate the average particle sizes and particles diameters of about 10, 15 and 25 nm were estimated from the powders prepared at 1100, 1350 and 1500 $^o$C, in that order. The lattice parameters in the largest grains were found to be about 4.330Å.

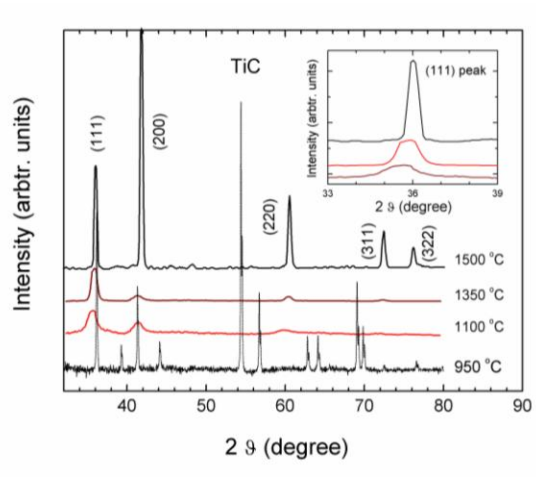

**Figure 1.** XRD pattern of sol-gel grown TiC particles.

## 3.2 Electrical Characterization

TiC pellets were used for electrical characterization. The CV tests were performed in 1 M HCl at 5-50mV/s scan rates with silver as reference electrode and platinum as counter electrode. As shown in Fig.2, different negative potential limit can be ascribed to the influence of pH on the hydrogen evolution reaction [7]. Small differences in peak height and width were observed for different electrodes in the same potential region. This may be attributed to the varying amount of carbon and oxygen as perfect stoichiometry is not always achieved in interstitial carbides. Any compositional variation or presence of vacancies, defects, pores, etc. can have a significant effect on the properties and behavior of these carbides [8]. The conductivity of TiC in the range of 30-900$^o$C is plotted in Fig. 3. The conductivity remains independent of temperature up to 600$^o$C and increases with further increase of temperature up to 900$^o$C showing a positive temperature coefficient for conductivity. The weak temperature dependence at low temperature indicates that the conductivity is entirely governed by the residual resistance of TiC. However, the possibility of charge carrier liberation with increasing temperature in semiconductors can also contribute to the increasing conductivity at higher temperatures.

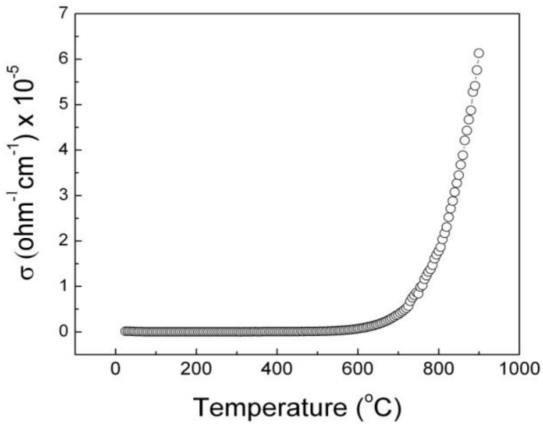


**Figure 3.** Conductivity variation with temperature in TiC.

## 4. References

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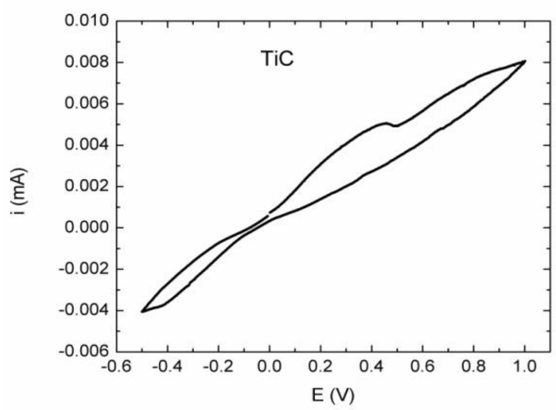


**Figure 2.** Cyclic voltammogram of TiC at 50mV/s.

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