

# Synthesis of $Ti_3SiC_2$ -Bicarbide Based Ceramic by Electro-Thermal Explosion

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## Abstract

A polycrystalline dense  $Ti_3SiC_2$  based ceramic material has been produced by several techniques. The effect of addition of TiC and SiC is also studied. The  $Ti_3SiC_2$  material shows extraordinary electrical, thermal and mechanical properties. Furthermore, it shows a damage tolerance capability and oxidation resistance. In this work, we have synthesized  $Ti_3SiC_2$  by electro-thermal explosion chemical reaction (ETE) with high current density (900 Amperes/a.u) followed by uniaxial pressure. The structural properties of the as-prepared materials are studied by x-ray diffraction (XRD), scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDX) techniques. The chemical cartography, imaging and electronic properties are investigated using Ultra-STEM and electron high energy loss resolution spectroscopy (EELS) techniques, respectively. The surface of  $Ti_3SiC_2$  is characterized by means of X-ray photoelectron spectroscopy (XPS). High resolution C 1s, Si 2p, Ti 2p, Ti 3s core level spectra are explained in terms of crystallographic and electronic structure. Valence band spectrum is performed to confirm the validity of the theoretical calculations.

## Keywords

$Ti_3SiC_2$  Based Ceramic, Intermetallic, Bicarbide, Electro-Thermal Explosion Reaction (ETE), Valence Band Spectra, Core Level Excitation

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## 1. Introduction

The investigations of the ternary ceramic  $\text{Ti}_3\text{SiC}_2$  show that it has exceptional properties [1]-[8]. Similar electrical and thermal conductivity at room temperature exceeds those of Ti metal; the former possesses excellent oxidation resistance (up to  $1400^\circ\text{C}$ ), a resistance to thermal shock; a moderately low coefficient of thermal expansion.  $\text{Ti}_3\text{SiC}_2$  also possess an unusual array of mechanical properties (including good compressive strength and a high Young's modulus all along with low hardness and some evidences for ductility) the ceramic is readily processed by standard tools. Also, it has been shown that large, highly oriented polycrystals of  $\text{Ti}_3\text{SiC}_2$  undergo auto-deformation plastically at room temperature by means of shear and kink band formation. The phase was first synthesized by Jeitschko and Nowotny back on 1967 [9]. Recent interest in the phase is due to the availability of improved material, which leads to high purity polycrystalline single-phase samples of near-theoretical density. The structure has been reported [10] as being hexagonal in space group P 63/mmc (194) with  $a < 0.307$  nm and  $c < 1.769$  nm. It shows a great resistance against oxidation, extreme hardness, and above all, it can retain its strength to temperatures that makes the best superalloys available today usable. Up to now, no other material has shown such a combination of properties like machinability, strength, and ductility at elevated temperatures and nonsusceptibility to thermal shock. It has an excellent self-lubricating property, so it can be better than graphite for rotating electrical contacts for ac motors. It is a promising candidate for ceramic engines. More details about the technological importance of  $\text{Ti}_3\text{SiC}_2$  can be found in reference [1].

As far as the experiments of this compound are concerned, Jeitschko and Nowotny [9] have synthesized  $\text{Ti}_3\text{SiC}_2$  via chemical reaction between  $\text{TiH}_2$ , Si, and graphite at  $2000^\circ\text{C}$ . Goto and Hirai synthesized  $\text{Ti}_3\text{SiC}_2$  through the chemical-vapor deposition technique [11]. Very little is known about this material. Panczyk *et al.* [10], shown that the melting point of  $\text{Ti}_3\text{SiC}_2$  occurs at around  $3000^\circ\text{C}$ . Pampuch *et al.* [12] produced a hard  $\text{Ti}_3\text{SiC}_2$ -based material with a high young's modulus of 326 GPa through self-propagating high-temperature synthesis and ceramic processing. Barsoum and El-Raghy [1] fabricated polycrystalline bulk samples of  $\text{Ti}_3\text{SiC}_2$  by reactively hot-pressing a blend of Ti, graphite, and SiC powders at 40 MPa and  $1600^\circ\text{C}$  for 4 h. Onodera *et al.* [13] [14] at high pressure measured the value of the bulk modulus at about 20666 MPa. The study of the electronic structure and chemical bonding gave a better insight into the important  $\text{Ti}_3\text{SiC}_2$  materials properties. In order to study the electronic structure of  $\text{Ti}_3\text{SiC}_2$ , full-potential linear-muffin-tin-orbital methods (FPLMTO) was used [15]. The calculations were based on the local-density approximation (LDA) and the Hedin-Lundqvist [12] parameterization was used for the exchange and correlation potential. Basis functions, electron densities, and potentials were calculated without any geometrical approximation [15]. The radial basis functions within the muffin-tin spheres are linear combinations of radial wave functions and their energy derivatives which are computed at energies appropriate to their site and principal as well as orbital atomic quantum numbers, whereas outside the muffin-tin spheres the basic functions are combinations of Neuman or Hankel functions [16] [17]. For the sampling of the irreducible wedge of the Brillouin zone, the special k-point methods were used [18]. Each calculated eigenvalue was associated with a Gaussian broadening of width 20 mRy.  $\text{Ti}_3\text{SiC}_2$  belongs to the D6h 4-P63/mmc space group of hexagonal crystalline structure [19], in which, its primitive cell contains two formula units. The observed lattice parameters were  $a = 53.064\text{\AA}$  and  $c = 517.650\text{\AA}$ .  $c/a$  was optimized by calculating of the total energy for different  $c/a$  values at the equilibrium volume. The calculated value of  $c/a$  was around 5.77 and it was in a very good agreement with the experimental value of 5.76. The calculated value of the bulk modulus was 225 GPa and is in a good agreement with the experimental value [17]. If one compares it with TiC, which has a bulk modulus of 240 GPa [20], one find that they are comparable; the density of states (DOS) for  $\text{Ti}_3\text{SiC}_2$  and the corresponding band structure is shown. The lowest-lying states around 10 eV are originated from the non-metal C 2s states. If one compares this with the DOS of TiC [21], there are also C 2s states which lie around 10.0 eV. Between 9.0 and 6.0 eV below the  $E_F$ , there are states which are derived from the Si 3s states. However, one can see a big gap in TiC in this energy range, due to the absence of Si. The states just below  $E_F$  are dominated by strongly hybridizing bonding states combinations of Ti 3d orbitals of eg symmetry, C and Si p-derived orbitals. The states above  $E_F$  contain antibonding Ti 3d-dominated orbitals of eg symmetry, C and Si p states. In the case of TiC, the states near  $E_F$  show the same character.  $E_F$  in TiC lies exactly in the pseudo gap, whereas in  $\text{Ti}_3\text{SiC}_2$  it has moved outside this gap. This indicates that TiC should be harder than  $\text{Ti}_3\text{SiC}_2$ .

In summary, it is shown that the modern electronic structure theory is sufficiently developed to give an accurate description of  $\text{Ti}_3\text{SiC}_2$ . The calculated volume and structural parameters are in a very good agreement with the experimental values. The chemical bonding appears to be similar to that of TiC.

A polycrystalline material based on a  $\text{Ti}_3\text{SiC}_2$  compound and produced by sintering the powders synthesized through the electro-thermal explosion (ETE) method has been shown to have the advantages of ceramic materials combined with some metal-type plastic properties. Reaction in ternary Ti-Si-C system under the ETE regime may produce either TiC, SiC,  $\text{TiSi}_2$ ,  $\text{Ti}_5\text{Si}_3\text{C}$  or  $\text{Ti}_3\text{SiC}_2$ : each in a quantity depending on the starting mixture composition [22]. However, until now, attempts to produce a  $\text{Ti}_3\text{SiC}_2$  single phase resulted at the very most in 10% TiC amount and traces of titanium-silicon intermetallic as ascertained. Polycrystalline dense  $\text{Ti}_3\text{SiC}_2$  based ceramic has been produced by several techniques and the effect of addition of TiC and SiC is also studied [23]-[33].

## 2. Experimental Procedures

Polycrystalline high-purity Ti, C, and Si powders (99.99% pure) of the nominal composition 3Ti/Si/2C, was mechanically alloyed. The powder was prepared from Goodfellow SARL powder in a high-energy Fritsch planetary ball mill with a balls/mass ratio of 13/1. The rotation speed could be varied within the range 450 - 650 rpm. In order to avoid oxidation during alloying, the ball mill was filled with high-purity argon gas. The vial was opened in 10 - 12 h to assure high homogenization and repeated fractioning. The powders were compacted into small discs (2 - 3- 13 mm) at the compacting pressure  $P = 6000$  psi and then subjected to ETE in Argon or in vacuum at  $900\text{ A}^\circ$  during 55 seconds followed by uniaxial pressure.

The structural properties were determined by XRD using a Philips diffract-meter (Co-  $K\alpha$  radiation). Further structural characterizations were carried out by energy-dispersive X-ray microanalysis (EDX), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), and optical microscopy (OM). SEM, FESEM micrographs were taken with a JEOL JSM-840A instrument equipped with an EDX accessory to check the elemental composition of the system [33]. XPS analysis were carried out in an ESCALAB Mk.II instrument using Al  $K\alpha$  X-rays as the source of excitation, the gun being run at 13 kV and 20 mA. Survey spectra were obtained over a kinetic energy range of 80 - 600 eV, in steps of 0.5 eV (CAE mode) of 100 eV. Detailed data were obtained for selected ranges of photoelectron energies (*i.e.* Ti 2p , C 1s , O 1s and Si 2p lines) in steps of 0.2 eV and at a pass energy of 20 eV; the regions were scanned repetitively in order to improve the signal-to-noise ratio. X-ray induced AES (XAES) structures data of Si KLL were obtained from excitation with Bremsstrahlung radiation; repetitive scans were carried out at pass energy of 20 eV and for steps of 0.2 eV. The XPS data were processed by subtraction of a Shirley-type background; minor charge shifting was accounted for by setting the graphitic adventitious C 1s component to 284.6 eV and by ensuring internal self-consistency of the shifted spectra. The noisiest of the spectra (e.g. Si KLL spectra) were smoothed, but not otherwise processed. The spectra from as-mounted, specimens were characterized by surface oxide and some adventitious surface carbon. Both were substantially removed by a light ion etch (ca. 6 mA  $\text{min cm}^{-2}$  dose of 3 keV) [6].

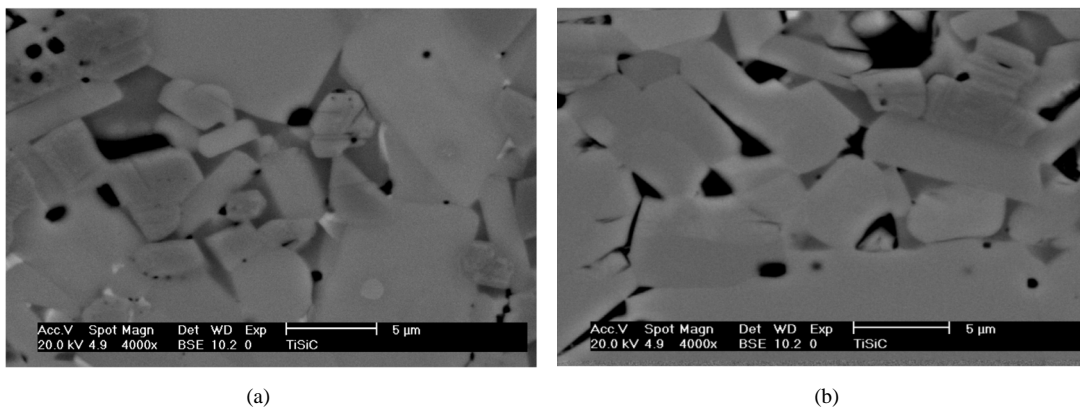
In this work, we have produced  $\text{Ti}_3\text{SiC}_2$  with chemical reaction by electro-thermal explosion with high current density ( $900\text{ A}^\circ$ ) followed by uniaxial pressure. The present study was examined the structural properties of materials using XRD, SEM and EDX. The chemical cartography, imaging and electronic properties will investigated using Ultra-STEM and electron high energy loss resolution spectroscopy (EELS), respectively. The  $\text{Ti}_3\text{SiC}_2$  is studied by means of X-ray photoelectron spectroscopy. High resolution C 1s, Si 2p , Ti 2p ,Ti 3s core level spectra are inspected in terms of crystallographic and electronic structure. Valence band spectra will be performed to confirm the validity of the theoretical calculations.

## 3. Results and Discussion

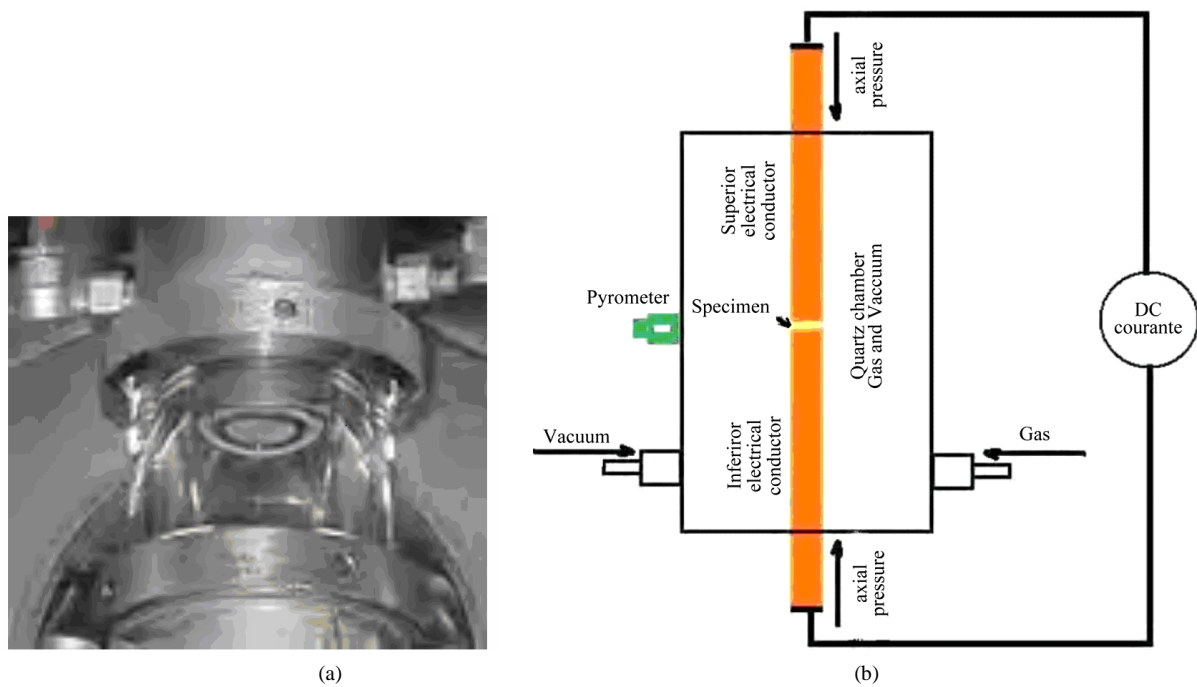
The final product has been investigated and analyzed by means of scanning electron microscopy (SEM), XPS, X-ray diffraction (XRD) and EDX. The observations have proved that the elongation slabs with rounded corners of well fused  $\text{Ti}_3\text{SiC}_2$  grains form a matrix within which some rounded TiC and less frequent angular SiC inclusions are present (Figure 1). Figure 1 shows the SEM images of the  $\text{Ti}_3\text{SiC}_2$ , bulkbicide base ceramic (TiC, SiC) produced via electro-thermal-explosion reaction (ETE, Figure 2) of crystalline elemental powder of Ti, C, and Si after balls milling (13/1 (balls/mass)) has been synthesized (Figure 3).

The phase composition after ETE reaction has been studied and analysed using the X-ray diffraction technique (Figure 4).

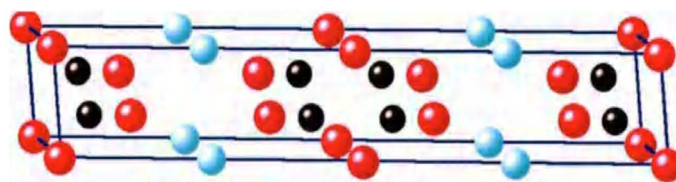
The morphology of the as-prepared materials was observed by scanning electron microscopy (SEM) as shown in the Figure 1. Further studies have been carried out by energy-dispersive X-ray microanalysis (EDX) (Figure 5)



**Figure 1.** SEM imaging: (a, b) representation of bulk  $Ti_3SiC_2$  corresponding to the carbides TiC, SiC and may be intermetallic  $TiSi_2$ .



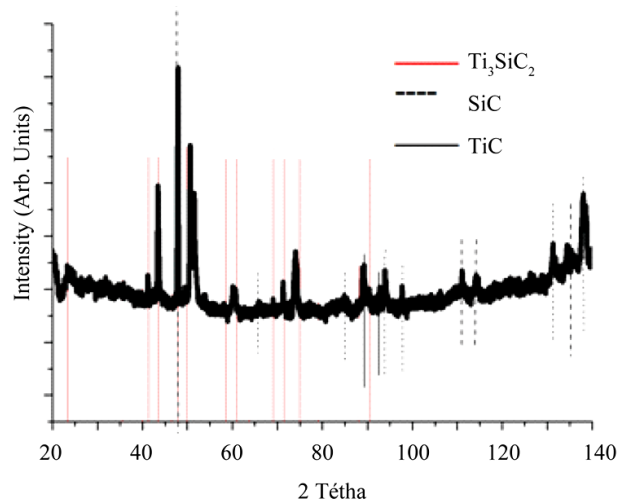
**Figure 2.** The electro-thermal explosion (ETE) reactor. (a) Chamber electro-thermal explosion reactor; (b) Schematic diagram of the electro-thermal explosion.



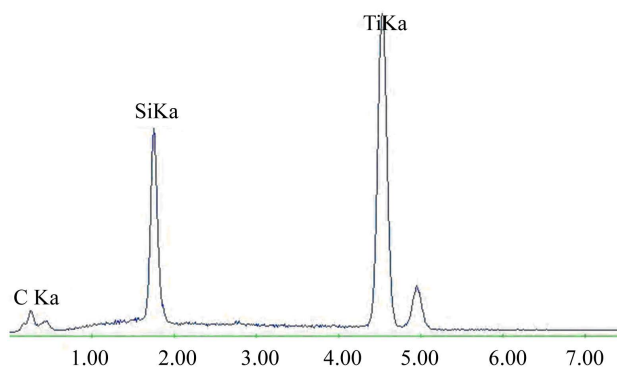
**Figure 3.** Crystal structure of  $Ti_3SiC_2$ , as refined from FPLO ab-initio calculations. Red, blue, and black spheres represent Ti, Si, and C atoms, respectively.

and X-ray photoelectron spectroscopy (XPS) (Figure 6).

The diffraction pattern of the combustion product formed upon ETE of the starting mechanically alloyed powder of Ti, C, and Si confirms the presence of  $Ti_3SiC_2$  bulk bicarbide base ceramic (TiC, SiC) and possibly



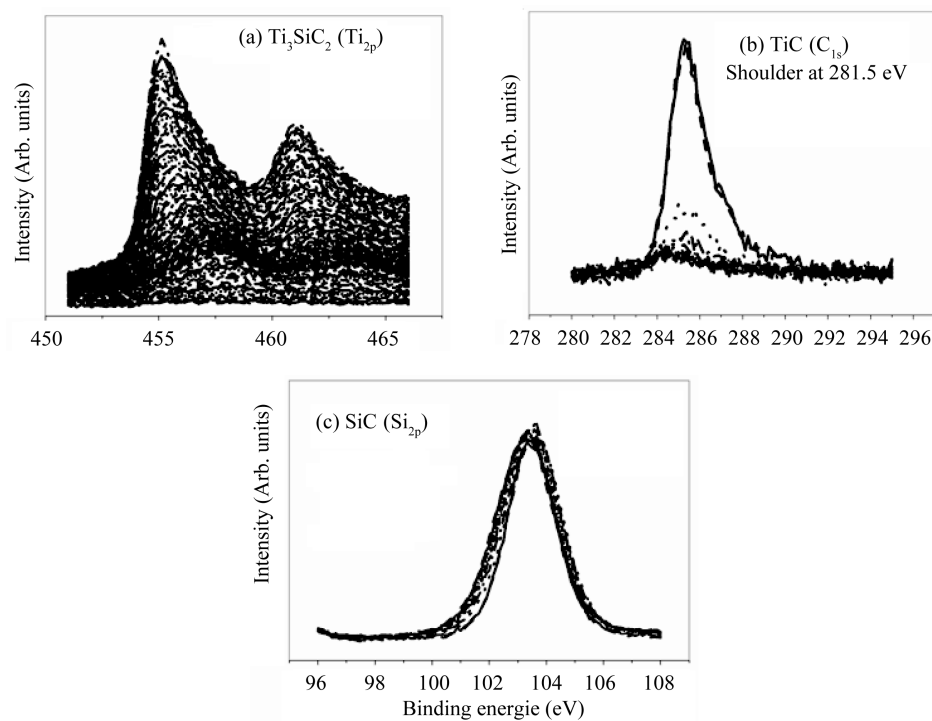
**Figure 4.** XRD patterns of the ETE from Ti-Si-C system product.



**Figure 5.** EDX spectra of bulk  $\text{Ti}_3\text{SiC}_2$  bicarbide based ceramic.

$\text{TiSi}_2$ . The morphology and microstructure of the ETE product are shown in **Figure 1**. The EDX data (**Figure 5**) also confirm the presence of the  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide base ceramic. The formation of the  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide is a two-step process: 1) the diffusion of Si into TiC to form the  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide, and 2) the diffusion of Ti into SiC resulting in  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide. The remaining amount of TiC and SiC could be subdivided in the matrix based ceramic.

The **Figure 6** shows the XPS data. The persistence of surface oxide is shown from the intensity of the O 1s line. The spectra of the model compounds were representative of those from the specimens that arise from optimized processing routes. Detailed spectra for the relevant specimens are shown in **Figures 6(a)-(c)** for C 1s, Ti 2p and Si 2p/KLL, respectively. There were a number of significant and less intense spectra, but the latter are all well resolved. The C 1s spectra (**Figure 6(a)**) for  $\text{Ti}_3\text{SiC}_2$  and (**Figure 6(b)**) for TiC revealed major contributions from adventitious graphitic/aromatic components at 284.6 eV, the carbide components were less intense, but well resolved at lower binding energies (BEs). The SiC spectrum, on the other hand, was dominated by a carbide peak at 283.5 eV and a small unresolved graphitic component on the high-BE shoulder of the main peak. The Ti 2p envelopes (**Figure 6(a)**) reflected the carbide contributions, as well as from the TiC surface that has the slightly higher BE. The detailed Si spectra (**Figure 6(c)**) exhibited various features. The SiC specimen resulted in envelopes (Si 2p and Si KLL) which are predominantly due to carbide, but with a small high-BE shoulder on the 2p envelope reflecting a SiOx state. The latter is matched by a resolved component in the KLL envelope at a kinetic energy (KE) of ca. 1608.5 eV. The spectra for  $\text{Ti}_3\text{SiC}_2$  exhibited two major contributions, one being at a BE of ca. 98.5 eV with a strong Si KLL component at ca. 1617.4 eV. And the second, which is most likely due to SiOx related features, that was found at ca. 102.3 eV with a matching KLL component at ca. 1610.6 eV. The error bars on the experimental results were typically 6, 0.2 eV, with somewhat larger uncertain-



**Figure 6.** High resolution X-ray photoelectron spectroscopy (XPS) spectra for: (a) Ti 2p, (b) C 1s and (c) Si 2p/Si KLL. The spectra refer to  $\text{Ti}_3\text{SiC}_2$ , TiC and SiC in order from the top to the bottom.

ties in the data for Si KLL. The present results for SiC and TiC are in excellent agreement with those from other studies. The C 1s low-BE component at 281.06, 0.2 eV for  $\text{Ti}_3\text{SiC}_2$  is close to the lowest C 1s value ever reported. Similarly, the Ti 2p<sub>3/2</sub> binding energy obtained from analysis of  $\text{Ti}_3\text{SiC}_2$  is close to that of metallic elemental Ti, *i.e.* 454.0 eV [6] [32], and is less, by ca. 0.5 eV, than that for TiC. Finally, the Si 2p binding energy for  $\text{Ti}_3\text{SiC}_2$  is below that of elemental Si, and some 2 eV is below those of the metal silicides [6] [32] and SiC.

The kinetic energy of the KLL transition is above that of elemental Si and is close to those for the metal silicides [6] [32]. The obvious qualitative conclusion is that the three constituent species are all exceptionally well screened (e.g., the Ti species appears to be as well screened as is in the metallic state), hence the low binding energies and the high Si KLL kinetic energy. The conclusion is in agreement with the observation that the ceramic is an excellent electrical conductor.

#### 4. Conclusion

We have successfully produced that the  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide based ceramic (TiC, SiC) via ETE-method. Further experiment will be focused on the synthesis of the pure  $\text{Ti}_3\text{SiC}_2$  bulk bicarbide with ETE and SHS reactions combined ETE-Sintering and ball milling-ETE. The aim of these investigations was to contribute to understanding the properties and to synthesis a single phase of these ceramic machinable materials.

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