Ultrahigh-pressure consolidation and deformation of tantalum carbide at ambient and high temperatures

Debrupa Lahiri a, Virendra Singh b, Giovani Ritta Rodrigues c, Tania Maria Haas Costa c, Marcia R. Gallas c, Srinivasa Rao Bakshi a,d, Sudipta Seal b, Arvind Agarwal a,*

a Plasma Forming Laboratory, Department of Mechanical and Materials Engineering, Florida International University, Miami, FL 33174, USA
b AMPAC and Nanoscience Technology Center, University of Central Florida, Orlando, FL 32816, USA
c Instituto de Física and Instituto de Química, Universidade Federal do Rio Grande do Sul, Porto Alegre RS, Brazil
d Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Madras, Chennai 600 036, India

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Abstract

The deformation mechanism of the ultrahigh-temperature ceramic, tantalum carbide (TaC), consolidated at room temperature at a very high hydrostatic pressure of 7.7 GPa is investigated using high-resolution transmission electron microscopy. The deformation behavior of TaC at room temperature is also compared with that consolidated at high temperature (1830 °C) at a similar pressure. TaC could be consolidated to a bulk structure (90% theoretical density) at room temperature. The deformation mechanisms operating at room temperature and 1830 °C are found to be significantly different. The room-temperature deformation is dominated by the short-range movement of dislocations in multiple orientations, along with nanotwinning, grain rotation, crystallite misorientation with low-angle grain boundary formation and lattice structure destruction at interfaces. In contrast, at high temperature, the strain is accommodated mostly by a single slip system, forming a parallel array of dislocations. The consolidation at room temperature occurs by heavy deformation with the support from short range diffusion, whereas the consolidation at high temperature is mostly diffusion dominated, indicating a classic sintering mechanism. The improved degree of consolidation with fewer defects results in significantly improved elastic modulus and hardness in the case of high-temperature consolidate.
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Keywords: Tantalum carbide; Ultrahigh pressure; Room-temperature consolidation; Deformation mechanism

1. Introduction

Tantalum carbide is an important transition metal monocarbide with an extremely high melting point of 3880 °C [1]. TaC has shown great promise as the next-generation aerospace material for throat and nozzle inserts due to its excellent refractory nature that can withstand the combustion flame temperature for most propellants [2]. However, the high melting point of TaC has made its consolidation into useful engineering shapes a major challenge. Achieving a fair degree of consolidation requires high-temperature exposure, as sintering occurs typically at temperatures >0.6Tm. The temperature required for consolidation is usually >2200 °C in pressureless conditions, whereas in pressure-assisted sintering (spark plasma sintering or hot isostatic pressing) it is >1200 °C [3–7]. Consolidation of TaC has been performed using different techniques, e.g. pressureless sintering, hot pressing, hot isostatic processing, high frequency induction heating, vacuum plasma spraying, spark plasma sintering and dynamic consolidation [8–21]. Additives like boron carbide, hafnium carbide, niobium carbide etc., have effectively reduced the sintering temperature to as low as 1400 °C, but at the expense of structural fineness and loss of mechanical performance [5,7]. Thus, there is a need for a suitable consolidation.
technique for TaC which can effectively decrease the consolidation temperature and retain the fine grain structure simultaneously.

The application of high pressure aids in consolidation through the powder metallurgy technique. Gallas and co-workers’ method of consolidating nanosized ceramic particles through the application of high pressure has shown great promise [22–24]. The application of high pressure (several GPa) can effectively consolidate ceramics such as alumina, silica and ferroelectrics, even at room temperature with a density >90%. In addition, these consolidates retained their nanosize grain structure and were free from secondary phase(s) from sintering additives. These studies inspired the present authors to explore the potential of a high-pressure compression technique in consolidating ultrahigh-temperature ceramics (UHTC), like TaC.

High-pressure consolidation of TaC at room temperature is expected to be deformation-dominated, considering the negligible chance of atomic diffusion. Thus, the high-pressure consolidation of TaC can possibly give new insight into the deformation mechanism leading to the manufacturing of an engineering component. The deformation behavior of TaC becomes different from other ceramics, due to the presence of a majority of Ta–Ta metallic bonds, unlike other ceramics, which have a majority of ionic and covalent bonds. The presence of a good amount of metallic bonds in TaC activates favorable slip planes and produces plastic deformation in a significant amount as compared to similar high-temperature ceramics, even at low temperatures [1,25–27]. However, these studies on room-temperature deformation of TaC are carried out using indentation based deformation and are limited to a very small volume [1,25–27]. The application of high hydrostatic compression over the entire volume of the body is expected to activate all possible deformation mechanisms to accommodate the severe plastic strain generated. Thus, it is possible to observe several possible low temperature deformation mechanisms in TaC over the large volume of high-pressure consolidate as compared to confined small volume beneath the indent.

In the context of the above discussion, the present study investigates an ultrahigh-pressure compression technique as a tool for room-temperature consolidation of TaC. The deformation mechanisms operating at room temperature and high hydrostatic pressure are analyzed with the help of high-resolution transmission electron microscopy (HR-TEM). Efforts are made to understand the consolidation mechanism of TaC at room temperature in terms of the structural changes taking place as a result of active deformation phenomena. A similar study was performed to consolidate TaC at high temperature (1830 °C) and ultrahigh pressure to understand and compare the deformation mechanisms operating at room and high temperature. The mechanical properties (elastic modulus and hardness) of TaC consolidated at RT and 1830 °C are also reported.

2. Materials and methods

Fine tantalum carbide powder, with an average particle size of $0.36 \pm 0.13 \mu m$, was obtained from Inframat advanced Materials LLC (CT, USA). The TaC powder has a purity of 99.7% with the total carbon content $>6.2~\text{wt.\%}$, free carbon $\leq0.15~\text{wt.\%}$, Nb $<0.3~\text{wt.\%}$ and O in the range of 0.13–0.3 wt.%. Fig. 1 shows an SEM micrograph of as-received fine TaC powders. The average particle size is $0.36 \pm 0.13 \mu m$.

Fig. 1. SEM micrograph of the as-received TaC powder. The average particle size is $0.36 \pm 0.13 \mu m$. 
pressure. After pressure stabilization, the sample was main-
tained at this pressure for 5 min. In the HP-HT experiment,
after pressure stabilization, the sample (without Bi gauge)
was heated to 1830 °C for 10 min. Quenching was per-
formed by turning off the power and, after 20–25 min, pres-
sure was released. Both the samples used in this study were
subjected to a similar hydrostatic pressure of 7.7 GPa, but
at ambient and 1830 °C, which will be referred as TaC-RT
and TaC-HT, respectively.

The density of the pellets was measured using helium gas
pycnometry (Accupyc 1340, Micromeretics Instrument
Corporation, Norcross, GA, USA) and expressed in terms
of relative density considering the true density of TaC as
14.67 g cm⁻³. The microstructural characterization was
carried out using a JEOL JSM-633OF field emission scan-
ing electron microscope operating at 15 kV. A Philips/
FEI Tecnai F30 high-resolution transmission electron
microscope, operating at an accelerating voltage of
300 kV, was used to analyze the lattice structure of the con-
solidated TaC. Forward and inverse Fourier transform
analysis, using Gatan, Inc. Digital Micrograph software,
was used for the accurate calculation of the lattice spacing.
ImageJ [30] software was used for quantitative analysis
from micrographs. Nanoindentation studies on both the
compacts were carried out using a Hysitron Triboindenter
TI-900 in quasistatic mode with a 100 nm radius diamond
Berkovitch probe. A peak load of 4500 μN was used for
indenting, with a constant loading/unloading rate of
450 μN s⁻¹ and a dwell of 3 s at peak load. Load vs. dis-
placement plots were obtained for more than 50 indenta-
tion tests on each sample over three randomly chosen
areas. The indents in each area were placed 9 μm apart to
cover a representative sample area for reporting the prop-
erties of consolidates. The Oliver–Pharr method [31] was
used to calculate the reduced elastic modulus ($E_r$) from
the unloading part of the load–displacement plot. The elas-
tic modulus ($E_s$) was calculated from the measured reduced modulus by introducing the correction factor for tip deforma-
tion using the following relationship:

$$
\frac{1}{E_r} = \frac{1}{E_s} + \frac{(1 - \nu_i^2)}{E_i}
$$

Fig. 2. (a) Schematic of the high-pressure consolidation system. Digital images of (b) TaC-RT and (c) TaC-HT pellets. The pellets were 1.5–1.9 mm in
thickness and 4.5 mm in diameter.
$E$ and $\nu$ are the elastic modulus and Poisson’s ratio, respectively. The subscripts $i$ and $s$ denote properties of indenter and sample, respectively. The Poisson’s ratio for TaC is 0.24 [32] and the elastic modulus and Poisson’s ratio for the diamond probe are 1140 GPa and 0.07, respectively [33].

3. Results and discussion

Fig. 2b and c presents a digital image of TaC-RT and TaC-HT pellets, respectively. It is interesting to observe that ultrahigh-temperature ceramic, like TaC, can be consolidated even at room temperature to a large, free-standing body, as observed in Fig. 2b. However, the pellet had broken into two parts during handling after the consolidation at room temperature. Table 1 summarizes the relative density, grain size and mechanical properties of TaC consolidated at room and high temperatures. The relative density of TaC-RT is $\approx 90\%$ without any sintering additive or second phase. The average grain size of TaC-RT is $0.42\, \mu$m, which is slightly higher than starting particle size of $0.36 \pm 0.13\, \mu$m. Thus, the room-temperature high-pressure consolidation of TaC is found to be successful in suppressing the grain growth. TaC-HT shows a higher relative density of $\approx 94\%$ with an average grain size of $0.50 \pm 0.10\, \mu$m. A 39% increase in grain size over the starting powder is obtained for TaC consolidated at high temperature.

The SEM micrographs of TaC-RT and TaC-HT (Fig. 3) reveal the difference in the consolidated structures. Fig. 3a shows a lower degree of consolidation with aggregate of TaC particles and the presence of porosity. TaC-RT does not show any sign of extended diffusion and neck formation as observed in SEM micrograph of TaC-HT (Fig. 3b). TaC-HT also shows a lower porosity, indicating higher degree of consolidation. The difference in degree of consolidation observed from microstructure is consistent with the density measurement by helium pycnometer.

Table 1 also presents elastic modulus ($E$) and hardness ($H$) values obtained from the nanoindentation experiments. The elastic modulus value shows a 150% improvement in TaC-HT (524 GPa) over TaC-RT (204 GPa). The nano-hardnesses of TaC-HT and TaC-RT are found to be 21.7 and 8.8 GPa, respectively, showing similar improvement again. The major contribution in improvement in $E$ and $H$ in TaC-HT comes from the degree of consolidation.

$E$ and $H$ are the elastic modulus and hardness, respectively. The subscripts $i$ and $s$ denote properties of indenter and sample, respectively. The Poisson’s ratio for TaC is 0.24 [32] and the elastic modulus and Poisson’s ratio for the diamond probe are 1140 GPa and 0.07, respectively [33].

Table 1. Effect of processing conditions on density, grain size, elastic modulus and nano-hardness of TaC consolidates.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Processing conditions</th>
<th>Relative density (%)</th>
<th>Grain size (µm)</th>
<th>Elastic modulus (GPa)</th>
<th>Nano-hardness (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TaC-RT</td>
<td>Ambient Temp., 7.7 GPa</td>
<td>89.8</td>
<td>0.42 ± 0.06</td>
<td>204 ± 22</td>
<td>8.8 ± 1.8</td>
</tr>
<tr>
<td>TaC-HT</td>
<td>1830 °C, 7.7 GPa</td>
<td>94.3</td>
<td>0.50 ± 0.10</td>
<td>524 ± 30</td>
<td>21.7 ± 1.5</td>
</tr>
</tbody>
</table>

Fig. 3. SEM micrographs of (a) TaC-RT and (b) TaC-HT revealing the difference in degree of consolidation.

However, only a 5% increase in degree of consolidation may not be solely responsible for such an impressive improvement in modulus and hardness of TaC-HT. It is envisaged that crystalline structure of TaC-HT with significantly less density of defects may be responsible for its impressive improvement in the mechanical properties. High-resolution transmission electron microscopic (HR-TEM) observations are made on TaC-RT and TaC-HT to understand the substructure (defects, nanocracks) and correlate with the operating deformation and consolidation mechanisms.

3.1. Deformation and consolidation mechanisms in TaC-RT

Ideally, ceramics with a majority of covalent and ionic bonds do not show any room-temperature slip or plastic deformation. However, ultrahigh-temperature carbides of group IV and V elements show some metallic behavior, due to the metallic bond between atoms, which is also reflected in their electrical conductivity [26–27,34]. In fact, transition metal carbides have a majority of metallic bonds due to their atomic arrangement in cubic-rocksalt type crystal structure [27]. The existence of metallic bonds in the slip systems makes plastic deformation easy. Researchers have concluded $\{111\}(110)$ to be the preferred slip
system for TaC at low temperature deformation produced by indentation [1,25,27,35]. These studies have proven the significant dislocation activity in TaC at low and ambient temperature. The TEM image in Fig. 4a reveals a high density of dislocations in multiple orientations in TaC consolidated at room temperature. Such arrangement of dislocations in different orientations in a small region suggests slip on multiple planes [34].

High dislocation density results from the accommodation of high strain created by an extremely high hydrostatic pressure of 7.7 GPa over the entire volume. Other sources of dislocation in the structure could be the nanocracks and nanovoids created during high-temperature application. Fig. 4b presents a nanocrack emanating from the surface of the compact at an angle of 40° to the (111) plane. The ICDD PDF No. 03-065-0282 is used for information on the lattice spacing of TaC. Cracks are reported to nucleate in TaC due to dislocation pile-ups and interactions at intersecting slip bands, under compressive stress at room temperature [25,36]. Crack tips, again, act as a good source of further dislocation activities and nanoscale deformation. Grain boundary rotation, sliding and nanotwin formation can also take place at the crack tip to relax the stress [36]. Local stress concentration near a propagating crack can initiate intergrain sliding and immobile dislocations [36]. Thus, the presence of nanocracks in TaC-RT is a rich source of high dislocation density. However, the structure does not get totally disintegrated due to crack suppression under hydrostatic stress condition [26]. The hydrostatic compressive force does now allow the crack to open up and propagate by suppressing the necessary tensile stresses normal to the plane of the cracks nucleation.

Apart from high dislocation density, amorphous regions and disturbed lattice structure is another significant feature of TaC-RT compacts. Fig. 5a presents severely disturbed lattice layers on the edge. The interior structure also contains multiple amorphous regions surrounding nanocrystallites as marked by arrows (Fig. 5a). Disturbance in the lattice structure is created by the application of high

![Fig. 4. HRTEM images of TaC-RT showing (a) the high density of dislocations in multiple orientations and (b) a propagating nanocrack.](image)

![Fig. 5. HRTEM images of TaC-RT revealing (a) the disturbed surface layer and the amorphous region surrounding nanocrystallites and (b) nanotwins, low-angle grain boundaries and high-angle grain boundaries with amorphous layer.](image)
pressure that causes maxima of localized stress at the surface of compressed particles leading to fracture [37].

Other modes of deformation and the results of high-dislocation activity in TaC-RT are also observed in high-resolution lattice images. Region A in Fig. 5b evidences the presence of a nanotwin in the TaC-RT structure. The twinning occurs on the (111) set of planes and the twin angle is 65°, whereas the twin band is ~3 nm wide. Such nanotwins have been observed by other researchers as a result of deformation in nanocrystalline metallic materials [38–40]. However, Li et al. have noted a similar feature in metal–ceramic composite multiscale structures, which they consider as not a typical twin structure; rather, it is due to the presence of stacking faults or dislocation cores [37].

Other than twinning, the high-density dislocations can also align and form low-angle grain boundaries to divide the structure into nanocrystalline regions. Regions B and C in Fig. 5b present two such grain boundaries with the angle of mismatch in (111) lattice being 5° and 9°, respectively. The same micrograph also shows the presence of a high-angle grain boundary with (111) lattice mismatch of 45°. The high-angle grain boundary consists of a ~2 nm wide amorphous/disturbed region.

Fig. 6 presents some wedge-like features in the lattice image of TaC-RT, which are produced due to grain boundary twisting [37]. It is possible to find out the intergrain twisting angles from the Moiré fringes that are created due to the angular mismatch of lattice orientation. The twist angle \( \theta \) is related to the spacing of Moiré fringes \( D \) and lattice spacing of the twist plane \( d \) with the following relationship:

\[
D = \frac{d}{2 \sin \frac{\theta}{2}}
\]

where \( D = 5.8/6 \text{nm} = 0.97 \text{nm}; d_{(111)} = 0.256 \text{nm}, \) thus the twist angle calculated is 15°. This twisting in grain boundary is possible due to rotation of grains with respect to each other. The application of compressive stress, even at room temperature, leads to material flow by grain reorientation and relative sliding along intergranular grain boundaries in ceramics [41]. Nanograin rotation can take place in extreme conditions, i.e. high value of applied stress and plastic strain rate, which generates a high density of dislocation. The shear strain generated inside a small grain forms a dipole, which causes the grain to rotate in order to accommodate the plastic deformation through crystal lattice rotation [42,43]. Thus, the grain rotation in TaC-RT seems to succeed in forming nanocrystallites due to segregation of dislocations at low-angle grain boundaries. Grain boundary sliding and rotation can orient the grains in positions favorable for slip [44].

Hence, the room-temperature consolidation of an ultrahigh melting ceramic like TaC under an extremely high pressure of 7.7 GPa can be explained in terms of multiple mechanisms postulated based on the deformation behavior observed in HRTEM micrographs (Figs. 4–6).

(i) The presence of numerous entangled dislocation networks (Fig. 4a) in TaC-RT indicates plastic deformation in the structure. During plastic deformation, crystallographic planes slip against each other and new surfaces are created on powder particles with numerous broken bonds. The adjacent particles going through plastic deformation together would take advantage of dangling broken bonds at the surface and due to pressure such surfaces come very close to forming new bonds. Especially, the structure of TaC comprises many Ta–Ta metallic bonds, which are easier to form. Such bonding would help in the consolidation of TaC-RT.

(ii) Consolidation can also be caused by short range diffusion at interparticle surfaces. The amorphous/glassy structure created at particle surfaces due to high-pressure application (Fig. 5a) causes easier diffusion through already disturbed lattices with high internal energy. Disturbed amorphous or glassy structures at the particle–particle interface or grain boundary of ceramic structure may allow rapid diffusion through the glassy layer [45]. This is more evident from the presence of an amorphous layer at the high-angle grain boundaries only (Fig. 5a), which is possibly created by coalescence of particles. On the other hand, the low-angle grain boundaries are created within one particle due to high deformation and thus do not have a thick amorphous layer at the boundary (Fig. 5a). In addition, diffusion can also be caused by a temperature rise at the interface due to friction generated heat on colliding surfaces at high pressure. A simple calculation is carried out on temperature rise at the interface based on conversion of pressure to heat energy, which is absorbed at a very
thin layer (1 nm) at particle interface. The calculations reveal a 350 K temperature rise at the interparticle surface at 7.7 GPa pressure, the specific heat for TaC at room temperature being 45.75 cal g\(^{-1}\) K\(^{-1}\) [46]. A temperature of \(\sim 375 ^\circ\)C may not be sufficient for significant diffusion in high-temperature ceramics like TaC. But simultaneous application of very high pressure can introduce some amount of short range diffusion in TaC, even with a small increase in the temperature.

(iii) The third possible mechanism of consolidation in TaC-RT is the mechanical interlock at particle surfaces. High pressure assisted collision at particle surfaces can very well create partial breakage and increased roughness at the particle surfaces. These rough surfaces with hills and ditches can provide some degree of mechanical interlocking when they fall in exact fitting. The importance of topological interlock in the strength of ceramic structures is also reported by Krause et al. in free gelation processed compacts [47]. The debris generated by breakage at particle surfaces can also fill the interparticle voids, thus increasing the contact area and improving the degree of consolidation in the TaC-RT composite.

A surprising \(\sim 90\%\) TD density of TaC processed at RT is a result of these consolidation mechanisms acting simultaneously.

3.2. Deformation and consolidation mechanisms in TaC-HT

As compared to TaC-RT, the deformation and consolidation mechanisms observed in TaC-HT are significantly different. Fig. 7a presents the traces of dislocations, which are all aligned in a similar direction as compared to random orientation of the same in TaC-RT. TaC-HT is consolidated at 1830 \(^\circ\)C. It is easy for TaC to deform in its most favorable \{111\}/\{110\} slip system. A significant plastic strain in TaC above 720 \(^\circ\)C has been reported [25]. Thus \{111\}/\{110\} dominated slip in TaC-HT results in parallel orientation of dislocations.

A qualitative comparison reveals much lower dislocation density in TaC-HT (Fig. 7a) as compared to TaC-RT (Fig. 4a). This observation can be justified in terms of stress relaxation that may take place in TaC-HT. Annealing at \(T_m/2\) for 1 h (1940 \(^\circ\)C) can cause strongly recovered and partially recrystallized structure in TaC [1]. In the present study the temperature is slightly lower (1830 \(^\circ\)C) than \(T_m/2\) and time is much shorter. But the presence of extremely high hydrostatic stress (7.7 GPa) can significantly reduce the requirement for recovery and recrystallization in terms of temperature and time by accelerating the diffusion. A clear evidence of recovery and recrystallization is recorded in lattice images of TaC-HT, viz. decreased dislocation density (Fig. 7a) and perfect lattice structure with no sign of amorphous region at the edges (Fig. 7b). The absence of any amorphous region on the surface and interior of the TaC-HT structure (Fig. 7b) is possible due to accelerated diffusion in TaC-HT, which causes perfect crystal lattice formation. In addition, the high-angle grain boundaries in TaC-HT also contain a thin disturbed lattice layer (marked by arrows in Fig. 7b) as compared to the thick amorphous region in TaC-RT (Fig. 5b). This is possible due to the coalescence of particles in TaC-HT in classical sintering mechanism due to the high rate of diffusion at the interface. No traces of nanocracks or nanovoids are observed in TaC-HT. Nanocrack generation is suppressed at high temperature due to accelerated diffusion along the grain boundary [36]. Thus it is concluded that the formation of nanocracks is partially suppressed in TaC-HT, which also suppresses the nucleation of dislocation and nanotwin formation. However, in addition to low density parallel dislocations, high stress in TaC-HT structure introduces another kind of deformation mechanism, which is grain boundary twisting. Fig. 8a presents the sign of Moiré fringes in the TaC-HT structure. And calculations according to the procedure
described earlier, following Eq. (2) \((D = 5.9/5 \text{ nm} = 1.18 \text{ nm}; d_{(111)} = 0.256 \text{ nm})\) in this case reveals a grain boundary twisting of \(\sim 3^\circ\) in case of TaC-HT, which is five times lower than that in TaC-RT. This observation is justified due to a much lower density of dislocation in TaC-HT, which can cause grain rotation. However, due to the parallel nature of dislocations in TaC-HT, it is generally unexpected to form a dipole inside a grain that can cause grain rotation. Some localized defects might be the cause of stress concentration and grain rotation in TaC-HT. The increased grain size is another inhibitor of grain rotation in TaC-HT. The grain rotation rate is found to have an inverse relationship with grain size \([48]\). However, this phenomenon needs further observation.

The evidence from SEM micrographs clearly indicates that classical sintering mechanism has taken place in TaC-HT with accelerated diffusion at particle surfaces, neck formation and closure of porosity. The TEM micrograph of TaC-HT (Fig. 8(b)) also shows grain rounding, which is an indication of sintering at high temperature. The grain size of TaC-HT is also 24\% higher than that of TaC-RT (Table 1), which indicates sintering and grain growth in the former.

4. Conclusions

This study substantiates the application of high pressure (7.7 GPa) as an efficient technique for consolidating ultra-high-temperature ceramics like TaC, even at room temperature. The deformation of TaC under high pressure follows different mechanisms at room temperature and high temperature of 1830 °C. At room temperature, multiple orientation of the dense dislocation network indicates activation of several slip systems apart from the most favorable \(\{111\}\{110\}\) for TaC. In addition, nanotwin formation, grain boundary twisting and disturbance of lattice layers at interfaces are other signs of deformation at room temperature. The consolidation in TaC at room temperature is governed by high-pressure-generated physical and chemical bonding at the interface and limited diffusion through disturbed/amorphous interface lattice structure. In contrast, the consolidation at high temperature is achieved through diffusion across particle interfaces, leading to neck formation inside the particles. The deformation at high temperature is dominated by a favorable slip system, generating parallel dislocations. The structure remains crystaline except high-angle narrow grain boundaries. TaC consolidate at high temperature shows \(\sim 150\%\) higher elastic modulus and hardness than the room-temperature one, which is attributed to the high degree of densification and absence of nanocracks, and other defects in the TaC-HT structure.

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Fig. 8. HRTEM micrographs of TaC-HT consolidate revealing signs of (a) grain boundary twisting and (b) grain rounding due to diffusion driven sintering.