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Enhanced low-temperature impact toughness of nanostructured Ti

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Impact toughness is one of the major mechanical properties for structural materials. It is generally observed that in coarse-grained materials the impact toughness decreases with decreasing testing temperature. Here, we report that the impact toughness of nanostructured Ti processed by severe plastic deformation is enhanced at low temperatures of $-70\text{ }^{\circ}\text{C}$ and $-196\text{ }^{\circ}\text{C}$, a unique phenomenon that contradicts the observations in coarse-grained materials. The enhanced impact toughness is attributed to the increased strength and ductility of nanostructured Ti as well as smaller fracture dimples at lower temperatures. This result demonstrates the advantage of using nanostructured Ti in low-temperature applications. © 2006 American Institute of Physics. [DOI: 10.1063/1.2167800]

Structural components made of metals and alloys are often subjected to impact loadings during their service. High impact toughness is desired to prevent catastrophic failure during an impact loading. This is especially important at low service temperatures. Metals and alloys, including steel, Ti and Al, usually have lower impact toughness with decreasing temperature,¹ which may limit their applications at low service temperatures. Some alloys, e.g. low strength ferritic steels, also have a ductile to brittle transition temperature, below which the steels become very brittle. Historically, tragedies have happened due to the low-temperature induced brittleness. One example is the sinking of the Royal Mail Ship Titanic, which was attributed to the freezing sea temperature ($-2\text{ }^{\circ}\text{C}$) at which steel hull plate of the ship became brittle.² Therefore, it has been a dream of material scientists to maintain or even to increase the impact toughness of metals and alloys at low temperatures. Such a dream has not been realized in conventional coarse-grained metals and alloys.

Most nanostructured metals and alloys are reported to have high strength but low ductility.³ Both high strength and high ductility are required to have high impact toughness.¹ Therefore, high strength of nanostructured materials may not result in high impact toughness if they have low ductility. Recently, some nanostructured materials processed by severe plastic deformation (SPD) techniques have been reported to have both high strength and good ductility,^{4–7} raising a prospect that some nanostructured metals and alloys processed by SPD might have high impact toughness. Indeed, an Al–Si alloy has been reported to have significantly improved impact toughness after being processed by an SPD technique, equal channel angular pressing (ECAP).⁸

Several recent studies have shown that both the strength and the ductility of some nanostructured materials increase with decreasing testing temperatures and increasing strain rates.^{9–12} The question is whether such an increase in strength and ductility leads to an increase in the impact toughness with decreasing temperature. It is the objective of

this study to investigate the temperature dependence of the impact toughness of nanostructured Ti processed by SPD. Our results show that nanostructured Ti has improved impact toughness at low temperatures of $-70\text{ }^{\circ}\text{C}$ and $-196\text{ }^{\circ}\text{C}$.

Commercially pure Ti-containing impurities, including (wt %) 0.2% O, 0.03% N, 0.2% Fe, 0.06% Si, and 0.56% Al, were used in this study. The initial coarse-grained (CG) Ti stock has an average grain size of $15\text{ }\mu\text{m}$. The nanostructured Ti was produced by ECAP+cold rolling followed by low-temperature annealing. CG Ti rods with a dimension of 60 mm in diameter and 120 mm in length were first processed by ECAP route B_C for 8 passes at $400\text{--}450\text{ }^{\circ}\text{C}$,¹³ in which the work-piece billet is rotated by 90° clockwise around its longitudinal axis between adjacent passes.^{14–16} The Ti rod processed by ECAP was first machined to remove surface defects and then cold rolled to 80% cross section area reduction without intermediate annealing. A series of rollers were designed in such a way that the cross-section of the Ti rod became oval shaped after odd-numbered rolling passes and round shaped after even-numbered passes. The as-rolled Ti rod was annealed at $300\text{ }^{\circ}\text{C}$ for 1 h. It is known that such annealing leads to a rearrangement of dislocation arrays, which improves ductility while sacrificing very little strength.^{14,17}

Charpy impact test samples with a dimension of $10\text{ mm} \times 10\text{ mm} \times 55\text{ mm}$ were cut along the cold-rolled rod by electrospark. A 45° V groove was machined in the middle of the sample with a depth of 2 mm and a radius of 0.25 mm at the root. The tests were performed on a Dynamic Impact Tester MK 30 with a maximal impact energy of 30 kg m and an impact speed of 5 m/s. The value of energy absorbed by the sample was determined to an accuracy of $\pm 0.1\text{ kg m}$. Tests were performed at $100\text{ }^{\circ}\text{C}$, $70\text{ }^{\circ}\text{C}$, $20\text{ }^{\circ}\text{C}$, $-70\text{ }^{\circ}\text{C}$, and $-196\text{ }^{\circ}\text{C}$. Samples tested at $100\text{ }^{\circ}\text{C}$ and $70\text{ }^{\circ}\text{C}$ were preheated in a muffle furnace for 15 min, samples tested at $-70\text{ }^{\circ}\text{C}$ were precooled in alcohol bath which was in turn cooled by liquid nitrogen, and samples tested at $-196\text{ }^{\circ}\text{C}$ were precooled directly in liquid nitrogen. The time between taking a sample out of the furnace/cooling bath and actual impact was less than 5 s.

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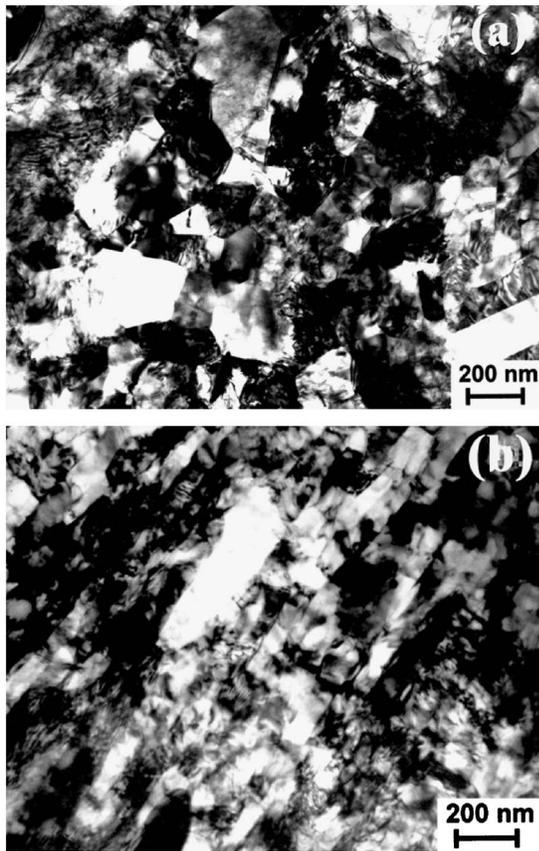


FIG. 1. Microstructures of nanostructured Ti in (a) cross and (b) longitudinal sections.

Figure 1 shows the transmission electron microscopy images of nanostructured Ti sample. Equiaxed grains were shown in the cross section [Fig. 1(a)] and elongated grains were shown in the longitudinal section [Fig. 1(b)]. In other words, the long axes of elongated grains are parallel to the longitudinal direction of the Charpy samples. Therefore, the crack propagation direction is perpendicular to the elongated grains during the impact. More details on the microstructures can be found in a previous paper.¹³

Figure 2 shows the impact toughness as a function of temperature for both nanostructured Ti and initial CG Ti. As shown, the impact toughness of CG-Ti decreases with de-

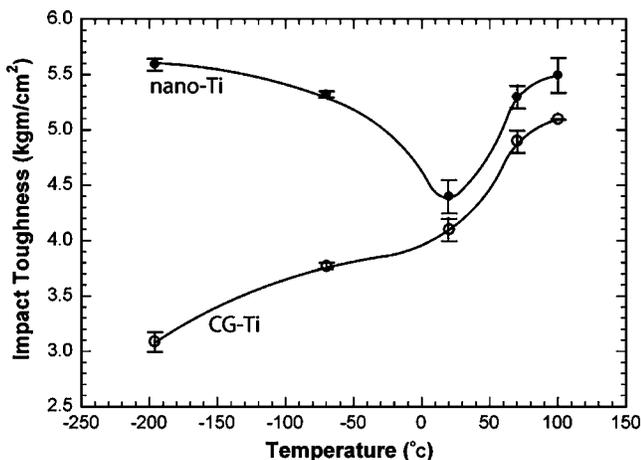


FIG. 2. The impact toughness of nanostructured and coarse-grained Ti vs temperature.

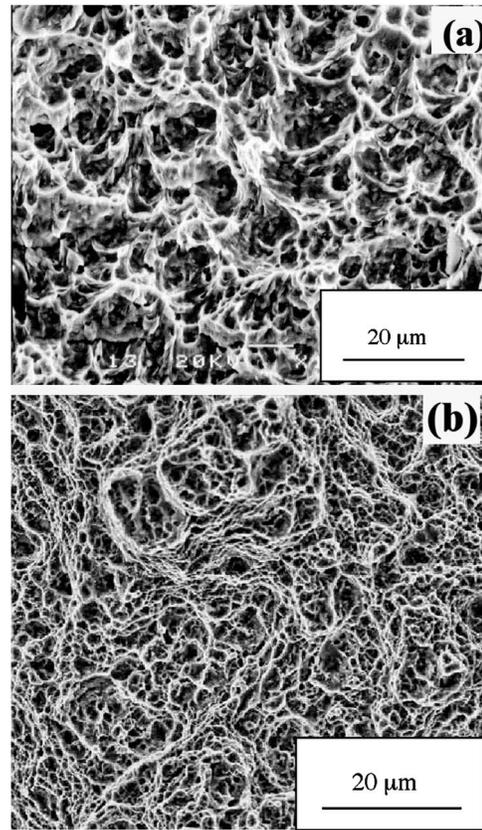


FIG. 3. SEM images of fracture surfaces of nanostructured Ti after impact test at (a) 20 °C and (b) -196 °C.

creasing temperature, which is consistent with observations reported in literature.¹ In contrast, the nanostructured Ti behaves very differently from the CG-Ti. In the temperature range from 20 °C to 100 °C, the impact toughness of nanostructured Ti follows a trend similar to that of CG-Ti, i.e., it decreases with decreasing temperature. Also, the nanostructured Ti has slightly higher impact toughness than CG-Ti, consistent with an observation on an Al-Si alloy,⁸ although not as dramatic. Significantly, below room temperature, the impact toughness of nanostructures Ti instead increases with decreasing temperature, a phenomenon that has never been observed before.

Figure 3 shows scanning electron microscopy images of fracture surfaces of nanostructured Ti after Charpy impact tests at [Fig. 3(a)] 20 °C and [Fig. 3(b)] -196 °C. Comparing Figs. 3(a) and 3(b) reveals that dimples formed at -196 °C are much smaller and have much higher density than those formed at 20 °C. The high density of small dimples requires much higher energy to produce than large dimples, rendering the nanostructured Ti a higher impact toughness when tested at -196 °C than at 20 °C.

The observed increase in impact toughness of nanostructured Ti at low temperatures can also be attributed to the simultaneous increases in both strength and ductility at lower temperatures and higher strain rates. The measured impact energy during a Charpy test consists of two components: The fracture initiation energy and the fracture propagation energy. A material with higher strength and higher ductility should have higher fracture propagation energy and, consequently, higher impact toughness. Nanostructured Ti processed by ECAP+cold rolling has been found to have higher strength and higher ductility at lower temperatures and

higher strain rates,^{6,9–11} which is attributed to its unique microstructures.

The nanostructured Ti has limited ductility at room temperature, due to its small dislocation cell size and grain size.^{6,18,19} During the deformation, the cell and grain boundaries can act as both dislocation sources and sinks, with dislocations bowing out of one segment of a cell/grain boundary and disappearing in another segment across the cell/grain. In other words, the defect generation and recovery are near a dynamic balance with no significant change in defect density, except at the initial stage of the deformation. Therefore, very little strain hardening exists after an initial short strain. Note that the nanostructured Ti tested in this study was annealed at 300 °C after ECAP+cold rolling. This annealing reduced the dislocation density through recovery, but did not affect the grain size.^{13,17} The annealing restored some work hardening but only to a limited extent. This lack of strain hardening makes the ligaments at the edges of the dimple fail quickly during the impact tests at room temperature and, therefore, it takes less energy for the fracture to propagate.

The nanostructured Ti has higher ductility at liquid nitrogen temperature.^{6,9–11,20} The saturation density of dislocations is determined by a balance between the dislocation generation rate and recovery rate, and this saturation density is expected to be higher at lower temperatures and higher strain rates.²⁰ Consequently, higher work hardening rate is restored and this leads to higher ductility.^{6,9–11,20} Both high strength and high ductility make it consume a large amount of energy for the ligaments of dimples to fracture. In addition, the fractographs shown in Fig. 3 indicates that more dimple ligaments needed to be broken at –196 °C than at room temperature, further increasing the energy consumption when the nanostructured Ti was tested at –196 °C. The higher strength, higher ductility, and larger amount of dimple ligaments are primary factors that contributed to the enhanced impact toughness of nanostructured Ti with decreasing temperature.

In summary, this work has demonstrated that nanostructured Ti processed by SPD has a higher impact toughness at temperatures much lower than room temperature. This observation is attributed to higher strength, higher ductility, and smaller dimple sizes of nanostructured Ti when tested at lower temperatures. It would be of interest to check if other

nanostructured materials processed by SPD have a similar behavior. The higher impact toughness with decreasing temperature makes nanostructured materials extremely attractive to cryogenic and other low-temperature applications. It should be noted that when the grain size is smaller than a certain critical value, the nanostructured metals and alloys could also exhibit a brittle fracture behavior,²¹ which makes it unsuitable to use nanostructured materials in service conditions involving impact loads.

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