

## **Influence of grain refinement on microstructural damage in DP1300 steel**

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**Abstract:** According to the Hall-Petch equation, the influence of microstructural refinement is well-known on the strengthening of metals and alloys. We have studied the effect of adding 0.14 wt% vanadium to a fine-grained (FG) DP1300 steel. The reference material has a mean grain size of 4.8  $\mu\text{m}$  in the rolling direction. Vanadium along with intercritical annealing cause remarkable grain refinement leading to an ultrafine-grained (UFG) DP1300 steel with a mean grain size of 1.6  $\mu\text{m}$  in the rolling direction. As a result, significant ductility improvement was achieved. This paper addresses the influences of grain refinement on the evolution of microstructural damage by means of optical microscopy, scanning electron microscopy and X-ray computed tomography. Results show that grain refinement reduces damage in the UFG steel, specifically during post-uniform deformation and leads to further elongation and necking before fracture.

### **1. INTRODUCTION**

Ferrite and martensite are the main microstructural constituents in dual phase steels. Although martensite has very limited ductility even ultrahigh strength dual phase steels exhibit ductile fracture due to nucleation and growth of voids. Initiation of voids in dual phase steels is associated with martensite cracking and decohesion of the ferrite/martensite interface due to plastic incompatibility of the two phases [1-3]. Ultrahigh strength dual phase steels have attracted much attention in the automotive industry due to their superior combination of strength and ductility, continuous yield point, good weldability and because of reasonable production cost. These properties make them strong candidates to enhance vehicle safety in case of an accident and also to reduce automotive weight. Obtaining tensile strengths beyond 1 GPa is possible in dual phase steels by increasing the volume fraction and carbon content of martensite. However, these methods lead to a reduction of ductility and crashworthiness. One alternative approach is through grain refinement. Song et al [4] has reviewed the processing, microstructure and mechanical properties of ultrafine grained (UFG) bcc steels.

### **2. BACKGROUND AND OBJECTIVE**

0.14 wt% of vanadium was added to a 0.23C-1.59Mn-0.19Si-0.03Cr DP1300 steel which led to grain refinement from 4.8 and 2.9 to 1.6 and 0.8  $\mu\text{m}$  in the rolling and normal directions, respectively. The Reference (Ref) and V-added DP1300 steels were processed by intercritical annealing at 750 °C for 120 s and at 760 °C for 90 s, respectively. After die quenching, martensite content was 40.5 $\pm$ 5.4 in the Ref and 40.2 $\pm$ 2.8 in the V-added steel. The processing and base-line mechanical properties of these materials have been reported by Scott et al. [5]. While true uniform elongation ( $\approx$ 0.08) and tensile strength ( $\approx$ 1500 MPa) remained similar in the 1mm steel sheets, area reduction based true strain to fracture increased from approximately 0.17 in the FG steel to 0.34 in the V-added UFG DP1300 steel. One reason for the ductility improvement was the suppression of micromechanisms of damage. This paper discusses influence of grain refinement on evolution of microstructural damage.

### **3. EXPERIMENTAL METHODS**

For metallography, samples were mounted, ground, and polished using 3 and 1  $\mu\text{m}$  diamond suspensions followed by 0.04  $\mu\text{m}$  silica suspension. 2% Nital was used for etching. Scanning electron microscopy was carried out using a JEOL 6610LV. Due to limitations of magnification and resolution, optical microscopy is not a sufficiently accurate method for quantitative analysis of voids in FG and especially UFG materials. Also, optical microscopy analysis is in 2D. Therefore, X-ray tomography was

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used for 3D quantitative analysis of damage. A Bruker Skyscan1172 X-ray computed tomography (XCT) scanner was used for quantitative analysis of microstructural damage. Scanning was conducted at 100 kV using an Al/Cu filter at 0.7 $\mu$ m pixel.

#### 4. RESULTS AND DISCUSSION

Adding 0.14 wt% V followed by the intercritical annealing treatment led to significant grain refinement in the DP1300 steel. This had no noticeable influence on uniform elongation and tensile strength. This was in fact expected; since the total carbon content, martensite volume fraction, and heat treatment cycle remained similar in the FG and UFG DP1300 steels. However, the post-uniform elongation and therefore ductility were significantly improved in the UFG V-added steel.

Our investigations show that the evolution of microstructural damage was very limited in both steels before the onset of necking. During post-uniform elongation voids initiated in the both steels primarily due to martensite cracking, and secondarily because of decohesion at the ferrite/martensite interface. Fig. 1 presents micrographs of the fractured steels subject to uniaxial tension. The black voids are seen more clearly in the as-polished bright micrographs at different locations (A, B, C and D) close to fracture surface. The microstructural locations of voids are shown in the etched SEM images. Images are presented at the same magnification for comparison. According to the optical micrographs, the two steels show differences as to the number, size, and area fraction of the voids, with more damage found in the reference steel. This can be related to the greater average martensite size and ferrite/martensite interface area which lowers the barrier for void nucleation and growth.

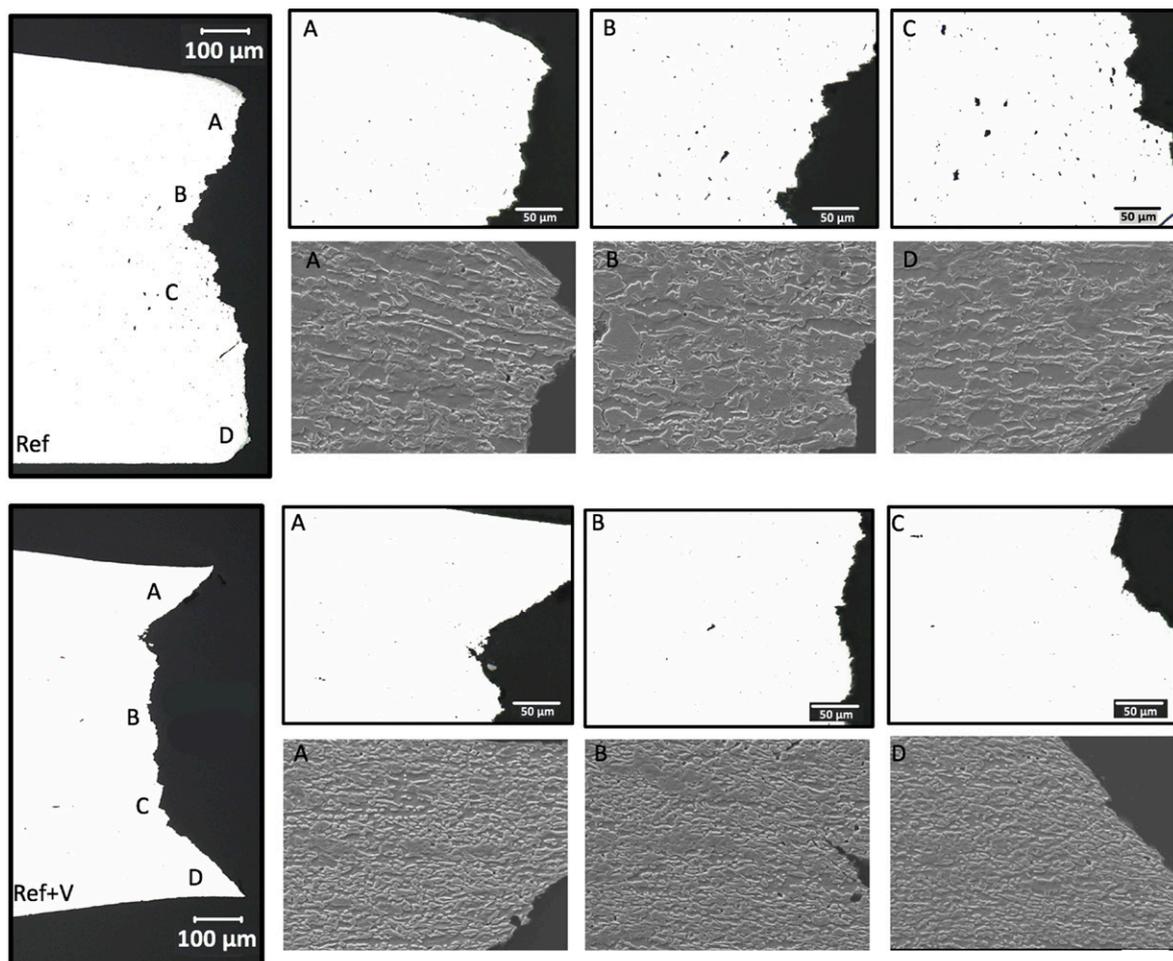


Fig. 1 Micrographs of the (top) Ref and (bellow) V-added DP1300 steels close to the fracture surfaces. Voids are shown in the as-polished bright and etched matrixes using OM and SEM, respectively.

Fig. 2 shows 3D X-ray tomography reconstructions of the Ref and V-added steels after deformation to fracture. The red colour represents voids inside the fractured samples. Most voids are located just below the fracture surfaces where localization of strain and stress triaxiality reach their maximum values.

Table 1 presents quantitative data obtained from XCT. Scanning of samples was carried out at the onset of necking and after fracture had occurred. The data set includes voids  $>15\mu\text{m}^3$ . The amount of damage is very small at the onset of necking; however, the number and volume fraction of voids in the FG steel are approximately four times larger compared to the UFG steel at this point.

The volume fractions of voids in both steels are similar at fracture. However, damage occurs more gradually in V-added steel. Therefore, to reach the final volume fraction of voids requires local strains of 0.17 and 0.34 in the FG and UFG steels, respectively. It seems that the critical void volume fraction for onset of fracture is about 0.3 vol% in the both steels and this amount was formed in the FG steel much faster than in the UFG steel during post-uniform plastic deformation. The number of captured voids in the FG and UFG steel was 3952 and 1901, respectively. This does not necessarily mean that the number of voids in the FG steel was almost two times higher; quantitative analysis is limited by resolutions such that only voids greater than  $15\mu\text{m}^3$  in volume (void diameter  $>2.5\mu\text{m}$ ) were taken into account. Since voids are generally smaller in the UFG microstructure, there may be many more voids that were eliminated from consideration in the UFG steel. That said, these small voids do not contribute in a significant way to the final failure process.

Although the microstructure was significantly refined in the UFG steel, the void mean diameter for the largest 50 voids was only slightly smaller in the UFG steel at fracture compare to that in the FG steel. It can be concluded that fracture occurred in the two steels after a sufficient amount of void growth and that this growth accrued in the FG at lower plastic strains compared to the UFG steels. The greater strain hardening in the refined microstructure of UFG steel could be partly responsible for the slower void growth.

Table 1. Summary of quantitative analysis of voids  $>15\mu\text{m}^3$

	Necked Samples			Fractured Samples		
	void number	void volume fraction %	void mean diameter* ( $\mu\text{m}$ )	void number	void volume fraction %	void mean diameter* ( $\mu\text{m}$ )
<b>Ref</b>	320	$4.35 \times 10^{-4}$	3.70	3952	$3.38 \times 10^{-1}$	6.51
<b>V-added</b>	73	$1.10 \times 10^{-4}$	3.32	1901	$2.97 \times 10^{-1}$	5.59

\* void mean diameter was calculated for the largest 50 voids

## 5. SUMMARY

Two DP1300 steels, one FG and the other V-added UFG, were subjected to uniaxial tensile tests. The uniform elongation and tensile strength were similar. Ductility however was much greater in the UFG steel due to improvement of post-uniform elongation. In this work, micromechanisms of damage were studied and 3D quantitative analysis of damage was carried out using X-ray computed tomography to investigate influence of grain refinement on microstructural damage. It was found that the rate of damage growth was notably lower in the UFG steel. This was due to the smaller size of the voids when nucleated: (i) smaller martensite particle size leads to smaller initial voids due to martensite cracking, and (ii) smaller ferrite/martensite interface area leads to smaller interfacial voids in the UFG steel. It is also well-known that strain hardening is enhanced by microstructure refinement. Consequently, slower growth of smaller initiated voids in the UFG steel leads to fracture at a higher strain.

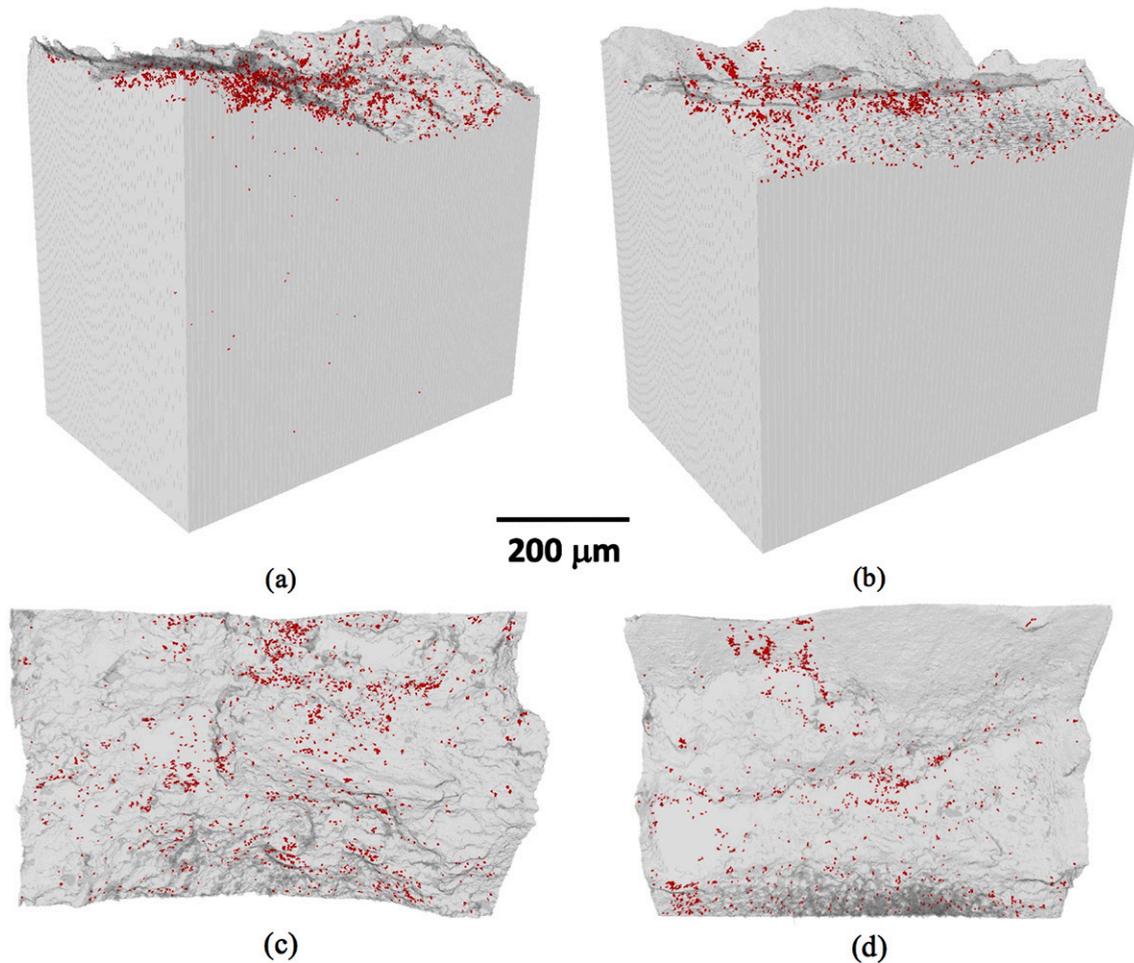


Fig. 2 3D X-ray tomography models of the fractured (a) Ref, and (b) V-added DP1300 steels. The normal view to the fractured surfaces are presented for (c) Ref, and (d) V-added steels. Voids  $>15\mu\text{m}^3$  are shown in red colour.

**Acknowledgements:** The authors are grateful to Colin P. Scott at CanmetMATERIALS, Hamilton, ON, Canada, who produced the DP1300 steels presented in this paper and made them available for this study.

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