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Research Article

Improvement in Mechanical Properties of Standard 15CDV6 Steel by Increasing Carbon and Chromium Content and Inoculation with Titanium during ESR

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The AFNOR 15CDV6 steel is high-strength steel with relatively low-level alloy content. In an earlier work, by processing the steel through ESR with inoculation, a marginal increase in strength and further increase in ductility and notch toughness were obtained. But the strength of the steel is inadequate for its use in fabrication of rocket motor casing in the Indian space programme. The present work aimed to increase the strength of the steel by increasing both carbon and chromium content of the AFNOR 15CDV6 steel at the expense of increased ductility and toughness due to processing through ESR. The increase in chromium content is expected to retard the bainite reaction resulting in an increased volume fraction of martensite in the mixed microstructure. Further, addition of chromium also causes secondary hardening during tempering. Another major objective was to study the effect of inoculation during ESR on grain size and mechanical properties. Titanium was used as inoculant in the present work.

1. Introduction

Ultrahigh-strength steels are becoming increasingly important in aerospace, defence, power generation, and in other applications/industries. The steel AFNOR 15CDV6 is a high strength bainitic steel containing low concentrations of chromium, molybdenum, and vanadium as alloying elements. Because of its good strength-ductility combination and ease of fabrication, the material has been extensively used in rocket-motor hardware in the Indian space programme. Sreekumar et al. reported that the microstructure of 15CDV6 steel in quenched condition consists of predominantly lower bainite and a small proportion of lath martensite [1]. The composition range and the mechanical properties of this steel in heat-treated condition are given in Table 1. However, the strength of this steel is inadequate for its use in larger rockets.

Earlier attempts to improve the properties of this steel through ESR resulted in large increase in ductility and toughness with little or no increase in strength [2]. A small increase in strength and further increase in ductility and notch toughness were obtained by inoculating the steel with 0.2% addition of niobium or zirconium during ESR [3].

A recent study by Young and Bhadeshia has reported that strength of mixed microstructure containing tempered martensite and bainite can peak at an intermediate volume fraction of tempered martensite of around 0.75 [4]. Increasing fraction of martensite in a mixed martensite-bainite microstructure can be achieved by the addition of alloying elements, which retard the bainite reaction. Alloying elements, particularly carbide forming elements, greatly retard the ferrite-pearlite reaction, and chromium, in particular, is very effective in retarding the bainite reaction. This often results in a bay of high relative austenite metastability being formed between ferrite-pearlite and bainite reaction whilst moving the whole TTT diagram to longer time. In view of the effectiveness of chromium in retarding the bainite reaction, it was expected that increasing the chromium content in the AFNOR 15CDV6 steel would lead to increase in strength.

Table 1: Chemical composition and mechanical properties of AFNOR 15CDV6 steel [2].

(a) Composition (wt%).

C	Si (max)	Mn	P (max)	S (max)	Cr	Mo	V
0.15	0.17	0.90	0.030	0.013	1.50	0.90	0.32

(b) Mechanical properties in the hardened and tempered condition*

UTS (MPa)	0.2% PS (MPa)	% Elongation	Charpy U-notch impact energy (kJ/m^2)	Hardness BHN
980–1175	830–980	8–12	650–700	360

* Austenitized at 980°C, quenched in oil, tempered at 650°C, quenched in oil.

Table 2: Chemical composition (wt%) of ESR ingot of Modified 15CDV6 at locations 2, 4, and 10 of Figure 1.

Sample location	Height (mm)	C	Si	Mn	P	S	Cr	V	Mo	Al	Ti
1a	30	0.27	0.12	0.65	0.042	0.015	4.02	0.26	0.68	0.038	0.060
1b	30	0.26	0.12	0.72	0.040	0.012	4.00	0.29	0.68	0.029	0.052
1c	30	0.27	0.15	0.67	0.040	0.010	4.12	0.26	0.65	0.037	0.040
4a	90	0.26	0.13	0.77	0.035	0.012	4.13	0.28	0.68	0.072	0.10
4c	90	0.26	0.15	0.75	0.038	0.010	4.10	0.27	0.65	0.067	0.07
10a	210	0.26	0.14	0.82	0.031	0.012	4.05	0.30	0.70	0.030	0.39
10b	210	0.27	0.13	0.83	0.034	0.010	4.20	0.30	0.70	0.058	0.40
10c	210	0.26	0.10	0.82	0.034	0.013	4.00	0.30	0.69	0.050	0.40

Accordingly, chromium content in the AFNOR 15CDV6 steel was increased from 1.5 to 4%.

As the strength level of steel increases, the defect tolerance level decreases. Therefore, a high degree of chemical and structural homogeneity and freedom from inclusions becomes important for ultrahigh-strength steels. For this reason, subjecting steel to a remelting process such as ESR becomes a necessity.

Promoting heterogeneous nucleation during solidification by inoculation technique refines the grain size and thereby results in improvement of strength and ductility. In order to promote heterogeneous nucleation, the substrate should have low contact angle and minimum lattice misfit with the nucleating phase. The substrate material may be added either directly or formed by in situ reaction in the melt. Both these methods have been applied during ESR. Campbell and Bannister [5] studied the effect of addition of oxides, carbides, nitrides, and borides of titanium, zirconium, and niobium during ESR of Fe-3% Si steel and observed columnar to equiaxed transition in the grain structure. The addition of TiO_2, TiC, and TiB_2 was found to be most effective in this respect. This method required the addition of very fine particle (10–100 microns) which is, however, not practical in large-scale ESR operation. Erdmann-Jesnitzer and Prozens [6, 7] and Chatterjee et al. [3] reported that additions of zirconium, niobium, titanium, and vanadium in the form of elements or alloys during ESR of steel result in significant refinement of grain size. While Erdmann et al. added inoculant continuously and directly into the slag bath, Chatterjee coated the electrode with the powder of ferroalloy using sodium silicate as the binder. In the present investigation a new technique of inoculation in situ ESR has been adopted by attaching to the electrode a thin-walled tube of appropriate diameter filled with requisite ferrotitanium powder [8].

The objective of the present work is to study the effect of grain size and compositional modification with respect to carbon and chromium on mechanical properties of AFNOR 15CDV6 steel. Carbon content in the steel has been restricted to 0.26% since its further increase will result in deterioration of both toughness and weldability of the steel.

2. Experimental Work

In the present work, electrodes for ESR were produced by melting mild steel scrap with additions of different ferroalloys to give the required composition in a medium frequency air induction furnace of 20 kg capacity. The liquid steel was cast into 40 mm diameter round in vertical chill moulds. These rounds were electroslag remelted using prefused slag of 70 CaF_2-30 Al_2O_3 compositions in a water-cooled steel mould of 80 × 70 mm crosssection using DC electrode positive power. The current and voltage during ESR were maintained at 1300 A and 32 ± 2 V, respectively. ESR ingot, was approximately 300 mm long and 12 kg in weight. Inoculation during ESR was carried out by attaching to the electrode a thin-walled mild steel tube of appropriate diameter filled with ferro-titanium powder. Carbon and chromium content of the ingot could also be increased, if necessary, by addition of high carbon ferro-chrome powder along with the inoculant.

Table 3: Mechanical properties of Modified 15CDV6 steel (0.26% C, 4% Cr, and 0.68% Mo).

Location*	Residual Ti content, (%)	Grain size, (μm)	Tensile properties UTS (MPa)	0.2% PS (MPa)	% El	% RA	Charpy U-notch impact energy (kJ/m^2)
2	0.050	31 ± 3	1430	1300	10	25	270
			1420	1280	09	23	260
4	0.085	26 ± 3	1480	1380	11	26	300
			1510	1400	10	30	310
10	0.39	18 ± 3	1580	1420	12	43	350
			1580	1400	11	43	340

* Austenitized at 960°C and tempered at 475°C.

The ESR ingot was homogenized at 965°C in a muffle furnace for seven hours. After homogenization, 20 mm and 10 mm lengths of ingot were discarded from the bottom and top of the ingot respectively. Samples were cut for chemical analysis as shown in Figure 1. Chemical analyses of the samples were carried out by the Polyvac vacuum spectrograph. Carbon was also analyzed by Strohlein coulometer. Titanium was analyzed both by ICP-AES and spectrograph.

Samples were heat-treated in a tubular furnace of constant temperature hot zone of 60 mm length in flowing nitrogen atmosphere to prevent oxidation. The temperature of the hot zone could be controlled within 5°C from the set temperature. The setup for heat treatment ensured a quenching time of less than 3 seconds.

The microstructures were studied using optical and scanning electron microscopes. For the purpose of grain size measurement, the austenite grain boundary was revealed by etching with Vilella's reagent.

For tensile tests, round specimens of 4 mm diameter and 20 mm gauge length were used. Tensile tests were performed on a 100 kN MTS machine and the Charpy U-notch toughness was measured on a standard Charpy testing machine. The fracture surfaces of the tensile specimens were examined in an SEM.

3. Results and Discussion

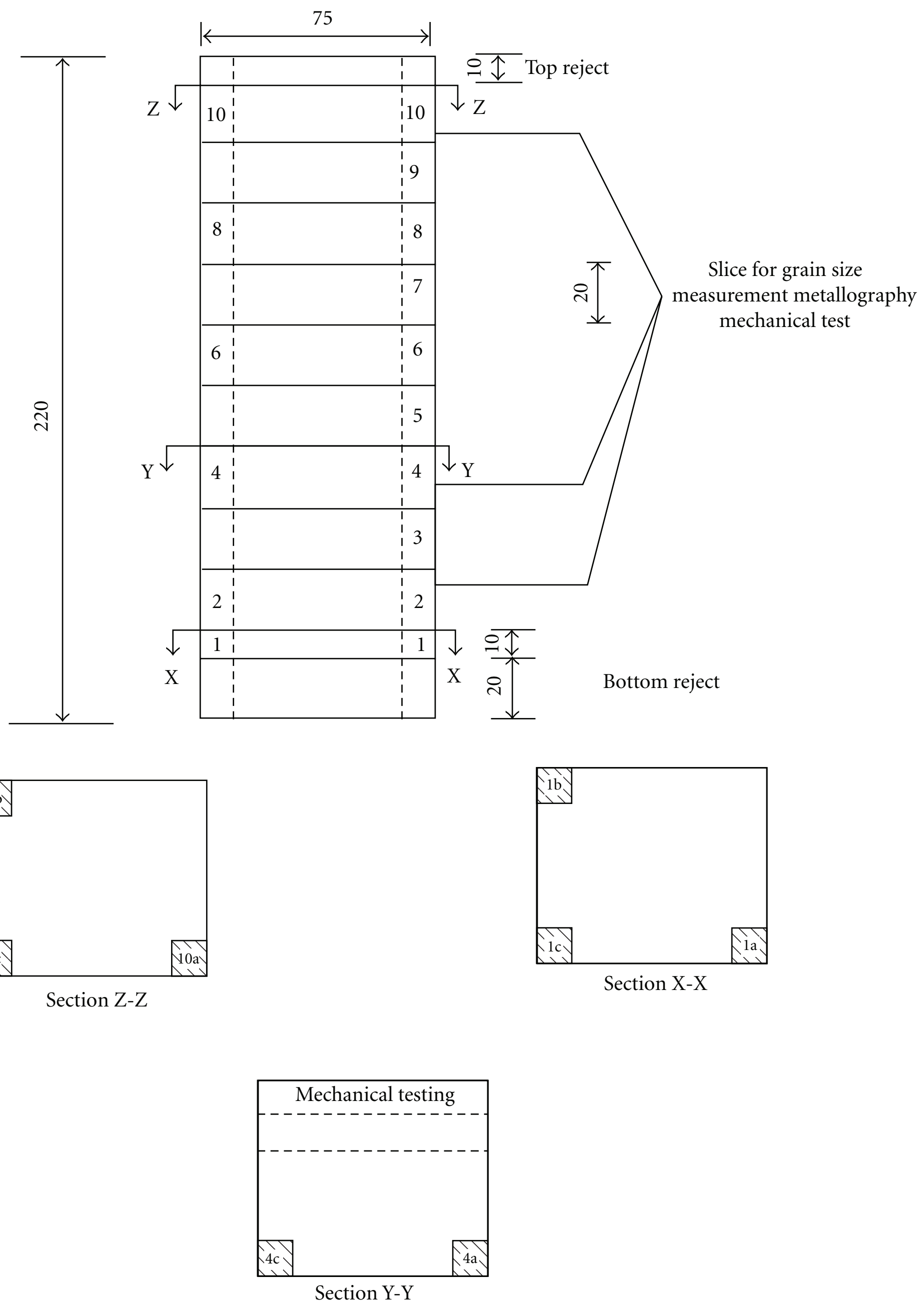
The ESR ingot obtained was free from defects such as porosity and cracks. The nominal compositions of the ESR ingot analysed at three different locations corresponding to heights 30, 90, and 210 mm, respectively, from the bottom of the ingot are shown in Table 2. It can also be seen from Table 2 that the composition of the ESR ingot was uniform along the heights of the ingot and for a given height the composition was found consistent at different transverse directions. The residual Ti content in the ESR ingot was found to be varying along the length of the ingot. This is due to the fact that during start up of the ESR the initial arcing was vigorous and the slag pool was yet to be formed resulting in heavy loss of titanium by oxidation. As the process gets stabilized it was carried out under a protective liquid slag cover resulting in high recovery of titanium almost 100%. At a height of 30 mm the average residual Ti content was found to be 0.050% whereas at the heights of 90 and 210 mm the residual Ti contents were analysed to be 0.085 and 0.39%, respectively.

From Figure 2 it is clear that the residual titanium content has a significant bearing on the grain size of the Modified 15CDV6 steel. For example, when the residual titanium content was analysed to be 0.050% (location 2) the corresponding estimated grain size of the steel sample obtained from the same location was found to be 31 microns. With further increase in residual titanium content to 0.085% (location 4) and 0.39% (location 10), the grain size of the steel at the corresponding locations was found to be decreased to 26 and 18 microns, respectively.

From Figure 3 it is clear that with increasing carbon and chromium content there has been increasing amount of martensite in the mixed microstructure of martensite-bainite (Figures 3(b) and 3(c)) of Modified 15CDV6 steel (0.26C, 4.00Cr, and 0.68Mo) as compared to the microstructure of AFNOR 15CDV6 Steel (0.15C, 1.50Cr, and 0.90Mo) consisting of predominantly bainite (Figure 3(a)).

In the case of uninoculated AFNOR 15CDV6 steel with estimated average grain size 37 microns, the ultimate tensile strength, ductility, and Charpy U-notch toughness were 990–1040 MPa, 17-18%, and 680–700 kJ/m^2 [3]. For the steel inoculated with titanium or niobium during ESR, the grain size was decreased to 15–17 microns, the yield strength was slightly lower (990–1020 MPa), but ductility and notch toughness increased to 18–20% and 860–920 kJ/m^2.

From Table 3 it is clear that the ESR Ti-inoculated Modified 15CDV6 steel with increasing carbon and chromium contents (0.26C, 4.00Cr, and 0.68Mo) showed the yield strength of 1280–1300 MPa at a grain size of 31 microns, an increase of almost 27–30% in comparison to inoculated ESR AFNOR 15CDV6 steel at a grain size of 17 microns. The yield strength of the ESR Ti-inoculated Modified 15CDV6 steel was further increased to 1380–1400 MPa and 1400–1420 by decreasing the grain size from 31 to 26 and 18 microns, resulting in increase in yield strength by 8 and 8–11%, respectively. The yield strength of the steel increased with decrease in grain size in accordance with the Hall-Petch equation (Figure 4(a)).



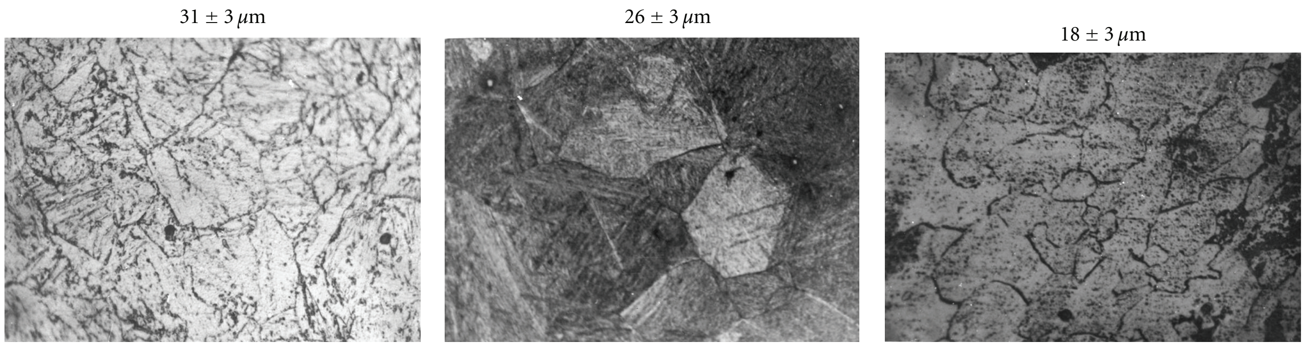
1a, 1b, 1c-10a, 10b, 10c: samples for chemical analysis
2c, 4c, 6c, 8c: sample for titanium analysis only
All dimensions are in (mm)

Figure 1: Sampling plan for the Modified 15CDV6 ESR ingot.

It is interesting to note that for an comparable grain size, that is, 18 microns, the yield strength of ESR Ti-inoculated Modified 15CDV6 steel (0.26C, 4.00Cr, and 0.68Mo), that is, 1400–1420 MPa, is almost 40% higher than that of the ESR Ti-inoculated AFNOR 15CDV6 steel (0.15C, 1.50Cr, and 0.90Mo), that is, 990–1020 MPa.

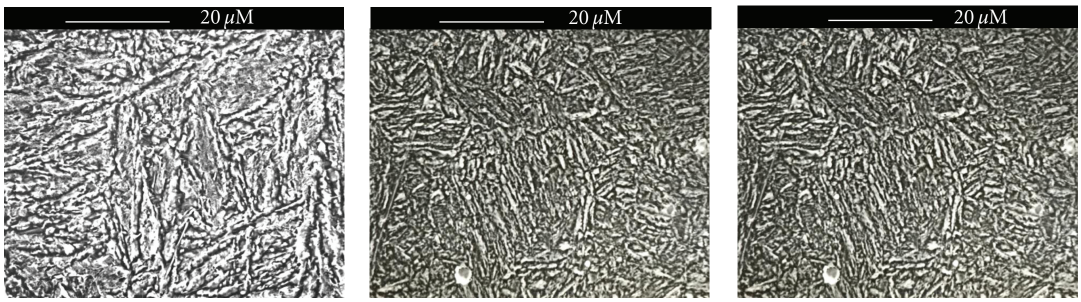
Like yield strength, the ultimate tensile strength of the ESR Ti-inoculated Modified 15CDV6 steel also increased with decreasing grain size. The ultimate tensile strength of the steel at a grain size of 31 microns was found to be 1420–1430 MPa whereas the strength was increased to 1480–1510 and 1580 MPa by refining the grain size to 26 and 18 microns respectively.

As expected the increase in carbon content from 0.15 to 0.26% decreased the Charpy U-notch toughness of the ESR Ti-inoculated Modified 15CDV6 steel but still it is within the acceptable limit. It is more important to note that refinement of grain size of the ESR Ti-inoculated Modified 15CDV6



(a) (b) (c)

Figure 2: Optical micrographs showing the grain structure of ESR Modified 15CDV6 steel at three different locations 2, 4, and 10 of Figure 1 in quenched and tempered condition. (×700). Etchant: Vilella's reagent.



(a) (b) (c)

Figure 3: The SEM microstructure of the ESR steel in quenched and tempered condition. (a) AFNOR 15CDV (0.15C, 1.50Cr, and 0.90Mo); (b) and (c) Modified 15CDV (0.26C, 4Cr, and 0.68Mo) at locations 4 and 10 of Figure 1. Etchant: 4% Nital.

steel (0.26C, 4.00Cr, 0.68Mo) from 31 to 18 microns resulted not only in an increase in strength but also in simultaneous increase both in ductility and toughness by 10 and 10–17% respectively (Figures 4(b) and 4(c)).

The fracture surface of tensile test specimens of the steel had a cup and cone contour (Figure 5(a)). The central interior region of the fracture surface had irregular fibrous appearance. The scanning electron micrograph of this region showed numerous dimples. The scanning electron micrograph of the fractured impact test specimen of the steel is shown in Figure 5(b). This shows that the fracture occurred in a ductile mode near the notch tip, indicative of a reasonable toughness.

This work clearly establishes that inoculation during ESR can significantly reduce the grain size leading to improved mechanical properties. The observed increase in strength due to increase in carbon content is well understood. The increase in strength with increase in chromium content can be explained as follows. From the micrographs it is clear that with increase in chromium content, the volume fraction of martensite has substantially increased. The increase in strength with increase in volume fraction of martensite has been attributed by Young and Bhadeshia [4] to two factors. When bainite forms, it enriches the residual austenite with carbon, so that the strength of the subsequent martensite increases. In addition, during its deformation, the strength of the bainite is enhanced via the plastic constraint by surrounding stronger martensite. An interesting feature observed is simultaneous increase in toughness and ductility with increase in strength at higher chromium contents. This is presumably due to precipitation of very fine uniformly dispersed chromium carbide particles, which resist the crack propagation.

4. Conclusions

(1) Addition of ferro-titanium through tube attached to the electrode during ESR is an effective method of inoculating steel with titanium.

(2) There is a clear relationship between the residual titanium content and the grain size of the inoculated ESR steel. The grain size decreases rapidly with increasing residual titanium content of the steel.

(3) Increase in carbon (0.15 to 0.26 wt%) and chromium content (1.5 to 4.0 wt%) of the AFNOR 15CDV6 steel leads to an increase in the martensite content in the mixed microstructure.

(4) The yield strength increases significantly with decreasing grain size of Modified 15CDV steel with increased carbon and chromium contents (0.26C, 4.00Cr, 0.68Mo). Interestingly, the % elongation and

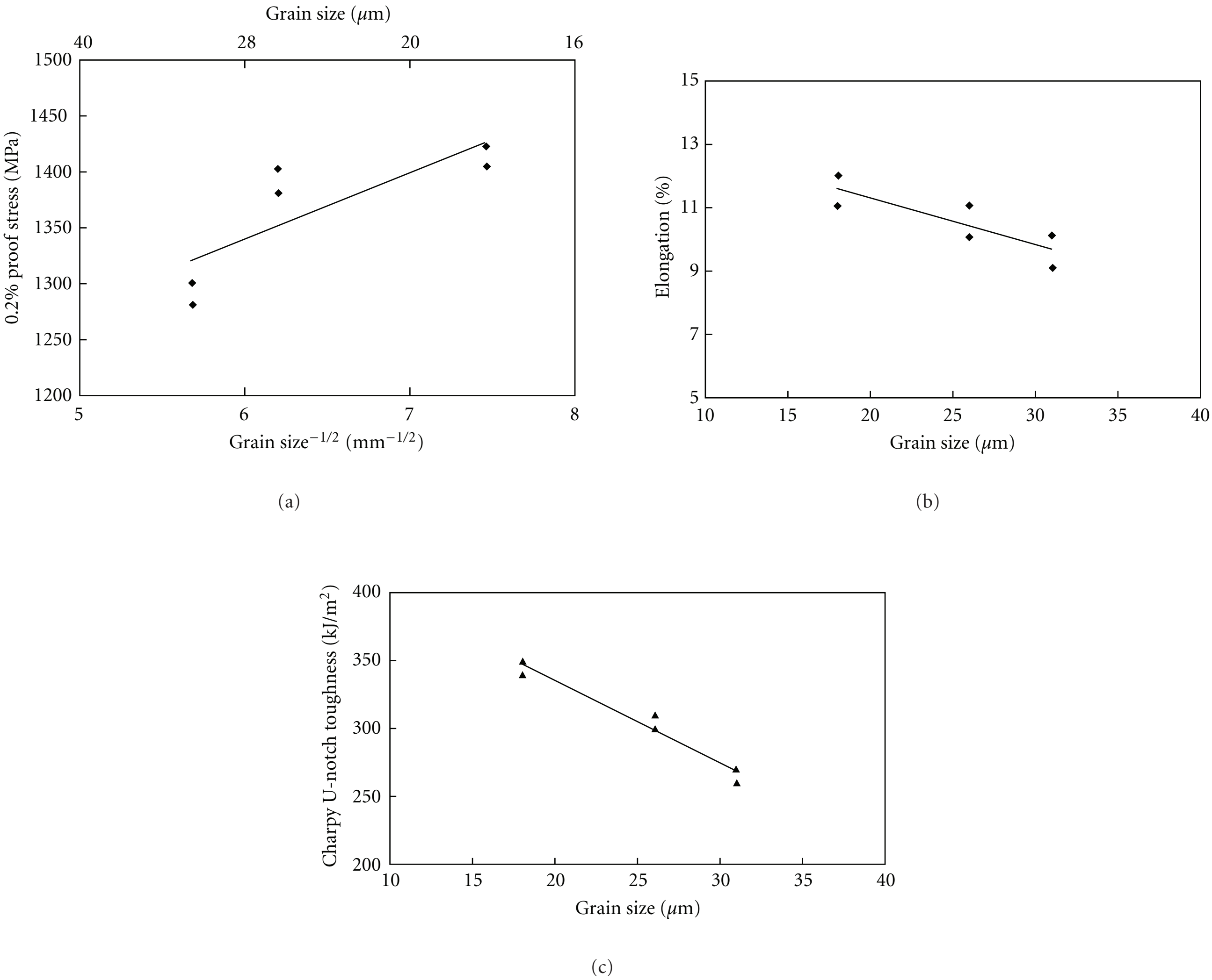


Figure 4: The effect of grain size on (a) 0.2% proof stress, (b) % elongation, and (c) Charpy U-notch toughness of Modified 15CDV6 steel (0.26C, 4Cr, and 0.68Mo).

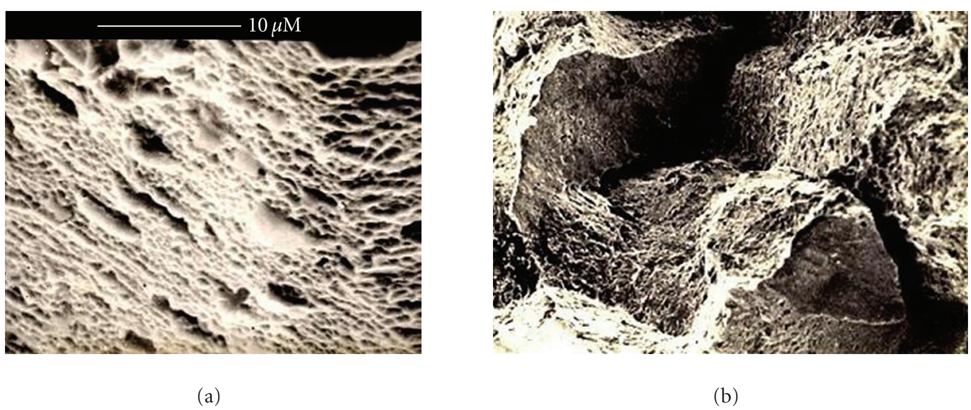


Figure 5: Scanning electron micrographs of fractured surfaces of Modified 15CDV6 steel (0.26C, 4Cr, and 0.68Mo), (a) Tensile specimen, (b) Charpy U-notch specimen.

Charpy U-notch toughness of the steel also simultaneously increase with decreasing grain size.

(5) The ESR Ti-inoculated Modified 15CDV6 steel (0.26C, 4.00Cr, and 0.68Mo) is capable of producing yield strength, % elongation, and Charpy U-notch toughness of 1580 MPa, 11-12, and 340–350 kJ/m^2 respectively.

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