

Optimization of BI test parameters to investigate mechanical properties of Grade 92 steel

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Abstract. The ball indentation (BI) testing is used to evaluate the tensile properties of materials namely yield strength, strength coefficient, ultimate tensile strength, and strain hardening exponent. The properties evaluated depend on a number of BI test parameters. These parameters include the material constants like yield slope (YS), constraint factor (CF), yield offset parameter (YOP). Number of loading/unloading cycles, preload, indenter size and depth of penetration of indenter also affects the properties. In present investigation the effect of these parameters on the stress-strain curve of normalized and tempered Grade 92 steel is evaluated. Grade 92 is a candidate material for power plant application over austenitic stainless steel and derives its strength from M23C6, MX precipitates and high dislocation density. CF, YS and YOP changed the strength properties considerably. Indenter size effect resulted in higher strength for smaller indenter. It is suggested to use larger indenter diameter and higher number of loading cycles for GRADE 92 steel to get best results using BI technique.

Keywords. Constraint factor, ball Indentation testing, , yield slope, indenter size effect , yield offset parameter,

1. Introduction

The ball indentation technique can be used to study the mechanical properties of steel including yield strength (YS), strength coefficient (K), ultimate tensile strength (UTS), strain hardening exponent (n), hardness and % elongation of the material at room temperature as well as at high temperatures. The system can be effectively used for determining remaining life of components in thermal and nuclear power plants. The technique requires very small material for testing and hence very useful; provided, it is standardized for particular material.

The BI technique consist of strain controlled multiple indentations on mirror finished and flat surface by spherical indenter. Associated penetration depth and the applied load (P) are continuously observed and recorded. The load-depth curve such obtained is then converted in to true stress-true plastic strain curve using standard empirical relations [1-4].

A detailed discussion on the conversion of load- depth curve into true stress true plastic strain curve by empirical relations has been reported by Mathew et al. [5].

Figure 1 (a) shows the deformed zone developed after loading and unloading of the indenter. 24% of



indenter radius is used to penetrate the material surface. 8 number of equal depth loading cycles each followed by partial unloading are used to attain this depth. h_t is the total depth and h_p is the plastic depth obtained after the elastic stresses are relieved once indenter is unloaded. Similarly d_t and d_p represent the total and plastic indentation diameter, respectively. The load-depth curve recorded is shown in Figure 1(b). This load depth curve is then transformed to true stress - true plastic strain curve by empirical relationships.

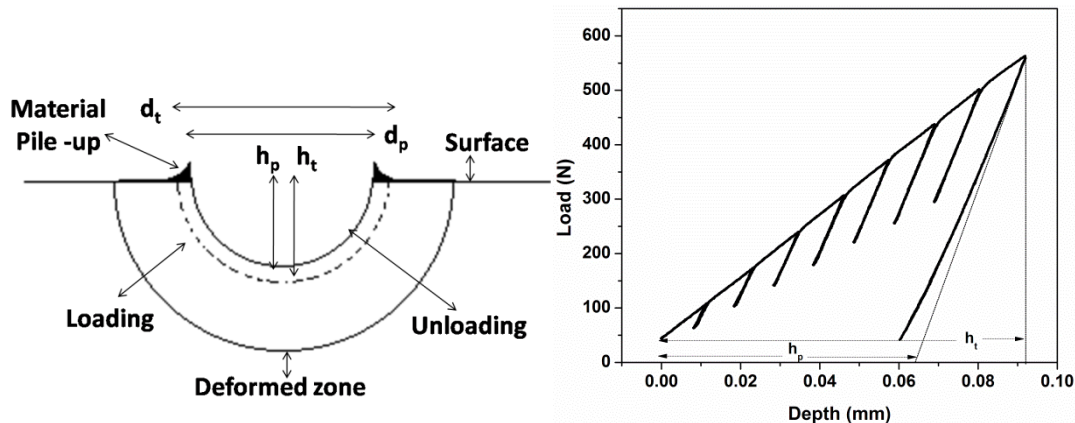


Figure 1. (a) Sketch of the Indentation obtained after Loading and Unloading, (b) Load-Depth Curve

In the present work, tensile properties of normalized and tempered GRADE 92 steels are obtained using BI technique at various test temperatures and results obtained are correlated with the results conventional tensile tests. The main objective of the work was to standardize the technique for GRADE 92 steel by studying the effect of various test parameters on the true stress- true plastic strain curve.

BI test parameters such as yield slope, constraint factor, yield offset parameter, indenter diameter, % of indenter radius used for penetration, frictional effect, strain rate etc. changes the tensile properties obtained from the ball indentation technique. Peter Miraglia has studied the effect of surface preparation, strain rate and indenter size on the evaluated tensile properties of samples prepared from A508 and A533B steels. He observed that the surface preparation didn't change the properties much but strain rate and indenter size have a significant effect on the properties. The changes had been compensated by adjusting the constraint factor and yield slope of the material while evaluating the properties [6,7].

GRADE 92 steel (9 Cr, 2W, 0.5 Mo) has been widely used for steam generator application in the nuclear and fossil fired power plants due to the high temperature strength, high thermal conductivity, and low thermal expansion coefficient offered by this material. GRADE 92 steel can be a substitute to austenitic stainless steels since it exhibits resistance to stress corrosion cracking and oxidation and exhibits good weldability [8-16]. GRADE 92 Steels exhibits tempered martensitic micro structure which is the reason of high strength offered by the materials. Adding to that MX and $M_{23}C_6$ precipitates present contributes to the solid solution strengthening and precipitation strngthening. Also they contribute to the transformation induced high dislocation density [10]. The microstructure of the GRADE 92 steel is significantly influenced by the heat treatment [8, 17].

2. Experimental Method

The chemical composition of GRADE 92 steel is given in Table 1. Spectro-chemical analysis method (ASTM-E-415-2008) is used for determining the composition.

Table 1. Spectro-chemical analysis of Grade 92 Steel

Element	Cr	W	Mo	C	Nb	V	Mn
Wt. %	9.4	1.9	0.5	0.1	0.07	0.2	0.39
Element	Cu	N	Al	B	P	Si	S
Wt. %	0.02	0.04	0.008	0.0018	0.01	0.02	0.004

To homogenize the microstructure of GRADE 92 steel plate, it has been normalized at 1323 K for 30 minutes followed by tempering at 1053 K for 2 hours and air cooled. This was the first step and steel was assumed to be as-received material. The steel plate is further cut in to blocks having dimensions of 20 mm x 20 mm x 12 mm, and 55 mm x 12 mm x 12 mm for BI and conventional tensile tests, respectively. Blocks were further normalized at 1353 and 1313 K for 30 minutes and tempered at 1013, 1033 and 1053 K for 1 hour followed by air cooling. Standard specimens were fabricated for conventional tensile and ball indentation tests from these heat treated blocks. The care has been taken to prepare the BI specimen having flat and parallel surfaces.

On the heat treated samples the tensile tests were carried out at strain rate of $3 \times 10^{-3} \text{ s}^{-1}$. In order to ensure uniform temperature throughout the specimen, the specimens were held for 15 minutes at test temperature prior to testing.

Further, in present investigation the efforts have been taken to study the influence of BI parameters on the tensile properties calculated from the BI test. The variation in the preload, yield slope, constraint factor, yield offset parameter, indenter diameter changed the properties significantly. Whereas the number of cycles and % radius used for penetration does not change the properties. A separate study has been carried out in order to lower down the gap in true stress- true plastic strain curve obtained between the tensile and ball indentation test.

3. Results and Discussion

3.1. The effect of BI test parameters on true stress – true plastic strain curve

The results obtained from the BI test basically depend on the different parameters used to carry out the test. The results were very sensitive to the material constants used like yield slope, constraint factor, yield offset parameter. These parameters were obtained in order to get accurate results. There were some other parameters like number of loading–unloading cycles, % of indenter radius used, which might affect the results. One important parameter was the indenter diameter used to carry out the test. In present study Silicon Nitride ball indenter of three different diameters were used to study the indentation size effect on the true stress-true plastic strain curve.

3.1.1 Effect of constraint factor, yield slope & yield offset parameter

As discussed before the constraint factor, yield slope and yield offset parameter are the material constants. The variation in the YS, UTS, K and n with the change in these parameters has been shown in Figure 2.

It was observed that as the constraint factor increased from 1 to 1.4, the UTS, K and n value decreased, but there was no significant effect on the calculated YS of the material. Since the constraint factor is a multiplication factor between the flow stress and mean indentation pressure, the increased constraint decreased the flow stress, resulting in decreased UTS, K and n value. The estimation of yield strength was not possible since the very low plastic strain associated with the yield strength was unattainable by any of the ball indenter used. The strain corresponding to first cycle was always above yield strain values. Thus using Meyer's hardness concept the yield strength was estimated. And hence change in the constraint factor didnot affect the yield strength values.

As the yield slope increased from 0.1 to 0.35, the K and n values decreased; there was an increase in the yield strength value with non-significant change in the UTS. The yield strength of the material is directly proportional to the yield slope and hence it increased with increase in the yield slope value. The increase in yield strength resulted in the decreased slope of the stress strain curve and hence the K and n values which are the output obtained from fitting this curve by Holloman equation decreased accordingly. The UTS is the result of the flow stress calculation as discussed above and hence UTS remains unaffected by change in the yield slope of the material.

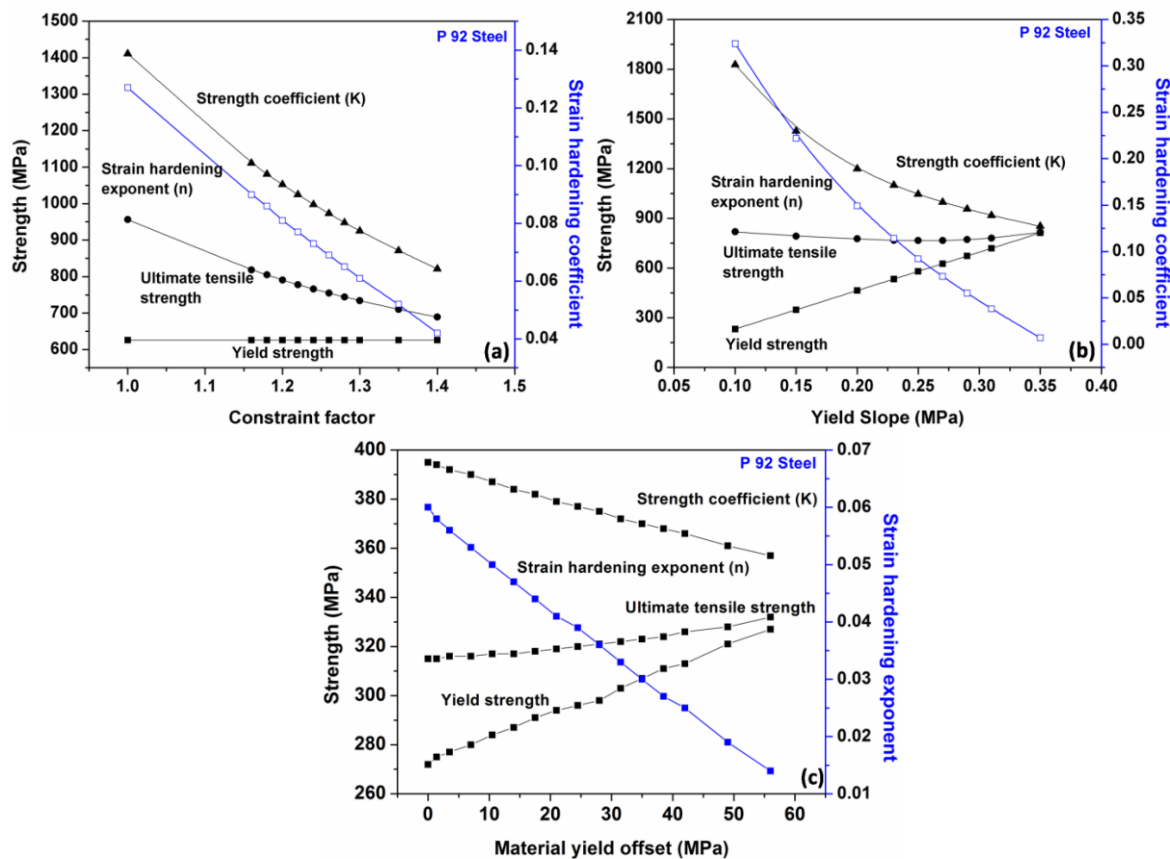


Figure 2. Variation in YS, UTS, K and n with Change in (a) Constraint Factor (b) Yield Slope and (c) Yield Offset Parameter

With increase in the yield off set parameter from 0 to 42 MPa, the properties showed the same trend as that of the yield strength. All the three parameters needed to be optimized in order to get the accurate results. The parameters have been obtained by regression analysis of the data obtained from the BI in order to match it with the data obtained from the tensile tests. The constraint factor and yield slope values finalized for the GRADE 92 steel are 1.32/0.24 for 0.25 mm, 1.24/0.28 for 0.76 mm and 1.08/0.32 for 1.5 mm diameter indenter.

3.1.2 Effect of number of cycles and % indenter radius used for penetration

Figure 3 shows the variation in yield strength and UTS values with respect to (a) change in the number of cycles and (b) % indenter radius used to carry out the test. It was observed that there was no significant change in the strength values with increase in the number of cycles and the % indenter radius used. The number of cycles only change the number of data points in the stress-strain curve. Since the load-depth relation was linear (Figure 1b) in case of GRADE 92 steel, the defined penetration depth got divided into number of cycles assigned, and hence there was no any change in the strength. In the same way the % indenter radius defines the maximum penetration depth and due to linear behavior of the curve there was no any change in the strength values.

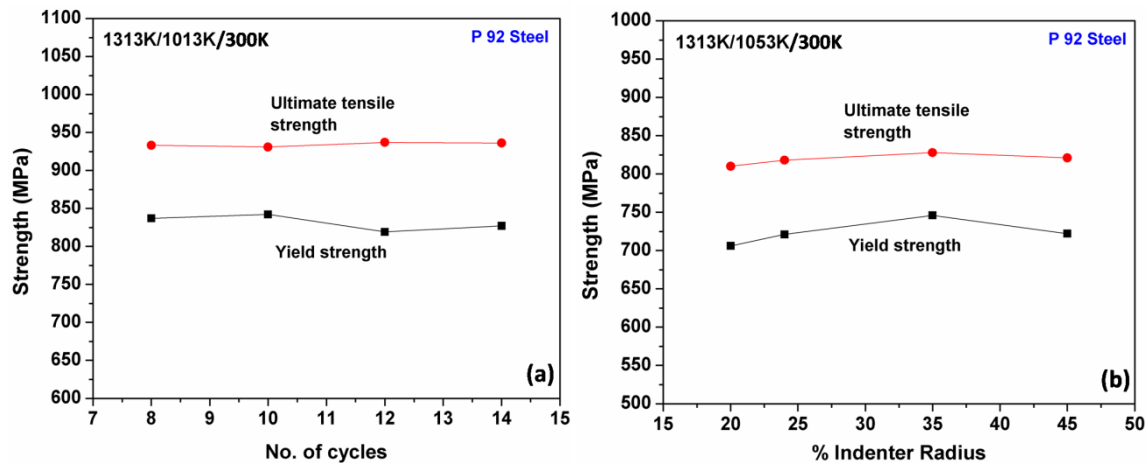


Figure 3. Variation in YS, UTS with (a) Number of Cycles and (b) % Radius Used for Penetration

3.1.3. Effect of indenter size

Figure 4 (a) and (b) shows the indenter size effect for the samples (normalized at 1353 K and tempered at 1013 and 1053 K) tested at 300 K. The BI test has been carried out using the silicon nitride ball indenter of diameter 0.25, 0.76 and 1.5 mm. It was found that as the indenter diameter increased the strength of material decreased. Thus the material constants i.e. constraint factor and the yield slope values needed to be redefined in order to get the accurate values of the strength (Figure 4 c, d). It was found that for smaller indenter the constraint factor was higher.

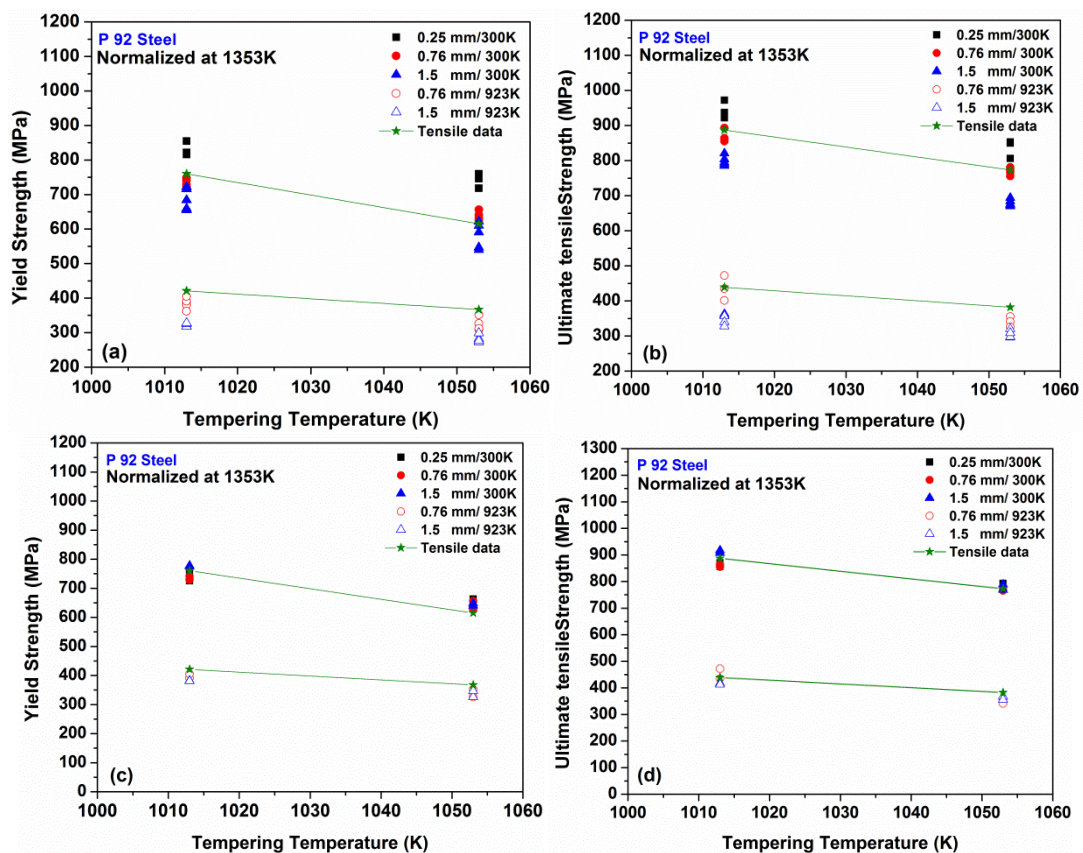


Figure 4. Variation in (a) YS, (b) UTS for Different Indenter Diameters, and Variation in (c) YS, (d) UTS for Different Indenter Diameters after Compensation with Yield Slope and Constraint Factor

This can be explained with the help of dislocation theory concept. Figure 5 shows the large and small indenters penetrated in to the material with specific load. The GRADE 92 steel consists of a large number of mobile and immobile dislocations. The plasticity occurred by the acceleration of pre-existing mobile dislocations or nucleation of the new mobile dislocations. The probability of the pre-existing dislocations available underneath the large indenter was higher as compared to that in case of small indenter and hence comparatively lesser load was required for large indenter to undergo plastic deformation. This has been explained by Shim et. al.[18]. The dislocations are separated by a distance called average dislocation spacing. As the diameter of the indenter changes, the high stressed region underneath the indenter also changes. If the radius of the indenter is smaller than the average spacing between the dislocations the probability of the availability of pre-existing dislocations in the region where high shear stress exists is very low. Thus for plastic deformation the higher applied stress is required to generate new mobile dislocations. In case of larger indenters the probability of pre-existing mobile dislocations underneath the indenter is very high in the stressed region and hence less energy is required for the movement of these dislocations.

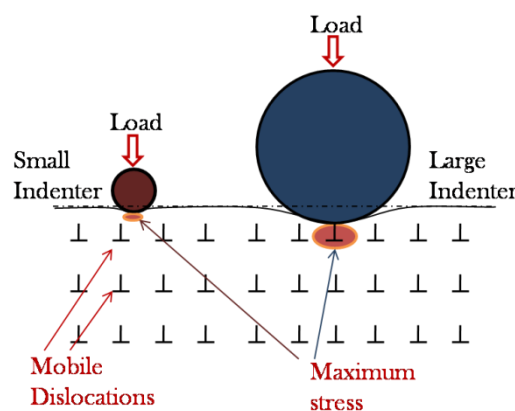


Figure 5. Schematic of Indentation Test for Different Sized Indenter along with the Array of Dislocations Present in the Material.

3.2. Optimization of parameters to reduce the gap between the results obtained from tensile and BI tests

The stress-strain curve obtained from conventional tensile test and the ball indentation test when superimposed, it was found that the data obtained from BI starts at the point where the stress strain curve from the tensile test ends.

The efforts have been taken to minimize this gap by optimizing some of the parameters. The range of strain covered in the BI test depends on the preload, number of cycles, indenter diameter used and the indentation depth.

The indenter diameter used defined the lower limit of strain and the upper limit is determined by the depth of penetration of indenter into the material [19]. The number of cycles used and the preload also shifts the curve a little towards lower strain. In this case, the upper limit of the penetration was set as 24% of the indenter diameter and the effect of all other parameters on the true stress-true plastic strain curve has been discussed.

3.2.1. Effect of preload

An initial test preload is applied on the sample surface for calculating the zero indentation point, on the BI load-depth curve before starting the actual test. Figure 6 shows the effect of preload on the true stress-true plastic strain curve obtained from BI test superimposed with the curve obtained from the tensile test. When the preload has been reduced from 44.4 MPa to 4.4 MPa, the stress strain data obtained from BI shifted towards the lower strain values and approached the tensile data. And hence it is advised to use smaller value of the preload while initiating the BI test. Indentation preload should be less than 10% of the indentation force at a depth value of 30% of the indenter radius [20].

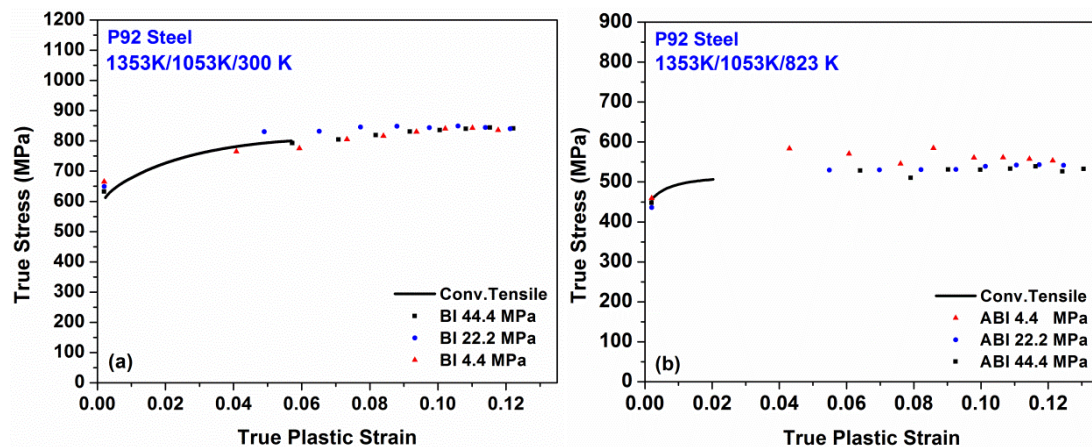


Figure 6. Effect of Preload on the True Stress - True Plastic Strain Curve Obtained from BI Test Superimposed with Tensile for Sample (a) 1353 K/1053 K/300 K. & (b) 1353 K/1053 K/823 K

3.2.2 Effect of number of cycles

Figure 7(a) shows the effect of number of cycles on the true stress- true plastic strain curve carried out on the sample heat treated at 1313 K/ 1013 K and tested at 823 K. It was found that as the number of cycles increased from 8 to 14 the true stress- true plastic strain data obtained from BI shifted towards the lower strain.

3.2.3. Effect of indenter size

Figure 7(b) shows the effect of indenter size on the stress strain data obtained from BI test. It was observed that as the indenter diameter increased from 0.25 mm to 1.5 mm the data points shifted towards the lower strain values. The data obtained for indenter diameter 1.5 mm diameter gave the better results among the three indenters.

In order to have good correlation between the BI test and tensile test results the number of cycles used should be on the higher end, the preload should be lower side, whereas for materials having lower ductility like GRADE 92 steel, the large indenter has to be used.

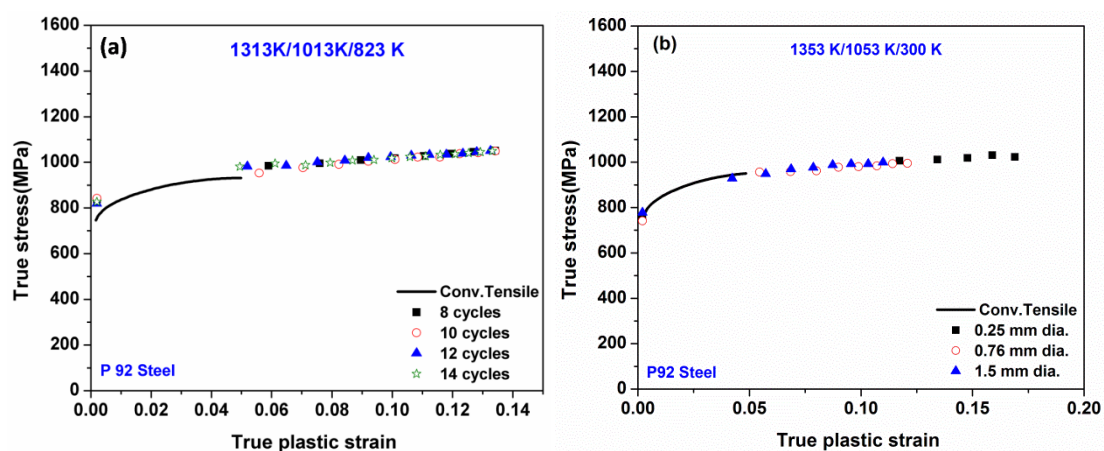


Figure 7. Effect of (a) Number of Cycles (1313 K/ 1013 K/ 823 K) (b) Indenter Size (1353 K /1053 K/ 300 K) on the True Stress - True Plastic Strain Curve (BI Test), Correlated with Tensile Test Results

4. Conclusions

GRADE 92 steel was subjected to heat treatment (normalizing (At 1313-1353 K) followed by tempering (At 1013-1053 K) and air cooled). And the Mechanical properties have been evaluated using conventional tensile tests and ball indentation technique. The effect of test parameters on the true stress- true plastic strain curve has been studied.

- It was found that as the indenter diameter increased the strength of material decreased. Thus the material constants i.e. constraint factor and the yield slope values were redefined as 1.32/0.24 for 0.25 mm, 1.24/0.28 for 0.76 mm and 1.08/0.32 for 1.5 mm diameter SiN indenter.
- In order to get best correlation between the BI test and tensile test results, the number of cycles used should be on the higher end, the preload should be lower side whereas for less ductile materials like GRADE 92 steel the large indenter has to be used.
- The BI technique can successfully be employed to investigate the mechanical properties of GRADE 92 steel.

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