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## Development of ultrafine bainite + martensite duplex microstructure in SAE 52100 bearing steel by prior cold deformation

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We explore microstructural refinement through complete/partial recrystallization of prior cold-deformed ferrite during austenitizing or austempering of SAE 52100 steel to obtain ultrafine bainite and martensite. Optical and scanning/transmission electron microscopy coupled with compositional microanalysis were employed to determine the volume fraction and dimension (thickness/length) of ferritic sheaves and carbides. Characterization of mechanical properties indicates that 5-15% cold deformation significantly enhances the impact strength ( $\sim$ 72 J) with marginal improvement in hardness and tensile strength over that in the undeformed condition.

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Conventionally quenched and tempered martensitic SAE 52100 steel has been the most commonly used material for ball and roller bearing applications [1]. The strength of the matrix coupled with a high level of primary alloy carbides imparts resistance to both abrasive as well as adhesive wear. However, extreme loading conditions demand a microstructure that can ensure improved mechanical properties of interest. The bainite-martensite duplex microstructure obtained through the austempering route is known to offer superior mechanical properties compared to those of quenched and tempered martensitic structure in SAE 52100 steel. The present study aims to further increase this scope by refining the sheaf thickness of relatively soft bainitic-ferrite interspaced between hard martensite plates/laths. Young and Bhadeshia [2] asserted that the presence of soft phases embedded in a hard matrix is conducive to enhanced mechanical properties. The conventional Hall-Petch relationship coupled with the effects of both substitutional and interstitial solid solution strengthening, work hardening and interprecipitate spacing is summarized by the following empirical equation [2]:

$$\sigma = \sigma_{\text{Fe}} + \sigma_c + \sum \sigma_{\text{ss}} + K_1 (L_3)^{-1} + K_{2\rho_d}^{1/2} + K_3 \Delta^{-1}$$
 (1)

where  $\sigma_{\rm Fe}$  is the strength of pure annealed iron,  $\sigma_{\rm c}$  is the contribution of solid solution strengthening due to carbon,  $\sum \sigma_{\rm ss}$  is the summation of solid solution strengthening due to all substitutional solutes,  $L_3$  is the ferrite plate thickness,  $\rho_d$  is the dislocation density,  $\Delta$  is the distance between the carbide particles (or precipitates) and  $K_1$ ,  $K_2$ ,  $K_3$  are constants.

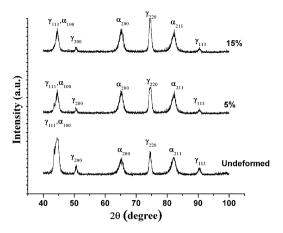
In order to reduce  $L_3$  in Eq. (1), attempts have been made to decrease the austenite grain size by prior cold deformation. Barford and Owen [3] and Umemoto et al. [4] have observed that the kinetics of solid-state bainitic nucleation increases when prior austenite grain size decreases. Graham and Axon [5] suggested that the growth of bainite is retarded when austenite grains are finer. Matsuzaki and Bhadeshia [6] reported that refinement of the austenite grain size leads to enhancement of the heterogeneous nucleation rate of bainite but the extent of the overall transformation is limited by a slow growth rate. In other words, lowering of prior austenite grain size may generate additional nucleation sites for bainite and enhance nucleation rate, but restrict the growth of ferritic sheaves by grain boundaries. However, refinement of ferritic sheaves and/or embedded carbides alone may not provide adequate strength. Thus, the present study aims to explore the scope of

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applying prior cold deformation to refine the prior austenite grain size and generate a duplex microstructure comprising submicrometer or ultrafine (<500 nm) ferritic sheaves together with martensite through controlled austenitizing and/or austempering followed by hardening/quenching.

Cylindrical samples 10 mm in diameter and 180 mm long of spheroidization-annealed SAE 52100 steel having a nominal composition of 1.1C, 1.46Cr, 0.27Si, 0.33Mn, 0.14 V, 0.04Ni, 0.02P, balance Fe (in wt.%) and showing an initial microstructure of spheroidized carbides in ferritic matrix, were subjected to tensile elongation of 5%, 10% and 15% in a universal tensile testing machine. The gripped end of the samples was discarded. The remaining parts of the samples were subjected to austenitizing at 950 °C for 15 min followed by instantaneous transfer to a salt bath for austempering at 270 °C for different time periods ranging from 10 to 70 min. The austempered samples were water quenched to room temperature. The austempered and quenched samples were mechanically polished with up to 0.1 µm diamond paste and etched with 2% nital (nitric acid in ethyl alcohol) for microstructural studies using optical microscopy and scanning electron microscopy (SEM). For transmission electron microscopy (TEM) studies, similar samples were mechanically thinned up to 90–100 µm thickness, and 3 mm discs were cut/punched from the thinned plates and subjected to twin-jet electropolishing using an electrolyte containing 15% perchloric acid and 85% ethanol at ambient temperature and 60 V potential (direct current). Mechanical properties, i.e. hardness, tensile and impact strength, of samples with appropriate dimensions and geometry were measured using a Rockwell/Vickers hardness tester, a universal testing machine and a Charpy impact tester, respectively. Analysis of phase aggregate (identity, volume fraction, crystallite size) and measurement of residual stress were carried out by X-ray diffracrtion (XRD) with a Panalytical X'pert PRO XRD unit using Cu  $K_{\alpha}$  radiation (0.154 nm).

Figure 1 shows the XRD profiles of the samples austempered at 270 °C/30 min without/with various degrees of prior cold deformation (5% or 15%). Evidence from the XRD pattern and optical microstructures suggests that the phase aggregate comprises ferrite, martensite, cementite and retained austenite. Table 1 shows the volume per cent of these phases determined from color tinted micrographs of the samples following austempering at 270 °C for various time periods (15–120 min) and water quenching without/with prior cold deformation



**Figure 1.** XRD profiles of undeformed + austempered (30 min), and prior cold-deformed + austempered (30 min) samples at various degrees of deformation, revealing different amounts of retained austenite.

(5%, 10%, 15%). It is apparent that the bainitic volume per cent increases, while both the austenite and martensite volume fraction decreases with increasing austempering time for samples without prior cold deformation. However, an opposite trend is observed in samples with increasing degree of prior cold deformation subjected to isochronal (30 min) austempering at the same temperature. It is anticipated that dislocation density increases with higher degree of cold deformation and leads to a reduction in the size of prior austenite grains by partial recrystallization during austenitizing at 950 °C for 15 min. It should be pointed out that prior deformation may be responsible for a greater degree of retention of austenite by mechanical stabilization, as seen in Table 1. Quantitative phase analysis on the basis of integrated intensity values of the (200) peak of ferrite (Fig. 1) as per the direct comparison method [7] yields the relative amount of phases formed for different degrees of prior cold deformation and austempering time (Table 1). It is relevant to mention that the amount of bainite marginally decreases with increase in the degree of cold deformation, mainly due to a significant reduction in the amount of prior austenite grain size by recrystallization prior to austempering and consequent restricted growth of ferrite within the small prior austenite grains [8]. The reduction in bainite volume per cent with the increase in the degree of prior cold work may also be due to the growth of ferritic sheaves being hindered by dislocation tangles remaining after incomplete

Table 1. Variation of relative amount of phases at different conditions of austempering time and degree of prior cold deformation.

Degree of prior cold deformation	Austempering time during austempering at 270 °C (min)	% Bainite	% Martensite	% Carbides	% Retained austenite	
					From microstructure	From XRD analysis
0 (undeformed)	15	10	77	1	12	
	30	35	52	3	10	
	60	40	47	5	8	
	120	42	48	5	5	
5%	30	32	61	2	5	6
10%	30	30	63	1	6	6.7
15%	30	25	63	2	10	9.2

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