Bulk nanoscale materials in steel products

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Abstract. Although a number of nanoscale metallic materials exhibit interesting mechanical properties the fabrication paths are often complex and difficult to apply to bulk structural materials. However a number of steels which exhibit combinations of plasticity and phase transitions can be deformed to produce ultra high strength levels in the range 1 to 3 GPa. The resultant high stored energy and complex microstructures allow new nanoscale structures to be produced by combinations of recovery and recrystallisation.

The resultant structures exhibit totally new combinations of strength and ductility to be achieved. In specific cases this also enables both the nature of the grain boundary structure and the spatial variation in structure to be controlled. In this presentation both the detailed microstructural features and their relation to the strength, work-hardening capacity and ductility will be discussed for a number of martensitic and austenitic steels.

1. Introduction
There is a very extensive literature on the production of nanoscale materials using a wide variety of techniques including vapour deposition, electro deposition, severe plastic deformation (SPD), ECAP and other methods. In a number of cases materials with very high strength and attractive combinations of strength and ductility can be produced. In addition the dominant length scales such as the grain size suggest are such that we need to modify the conventional descriptions of yielding and work hardening to account for the events which occur at these reduced length scales.

However few of these investigations yield materials which can be considered as bulk materials which can be readily utilized for current engineering applications. An alternative approach is to consider a more conventional material such as a heat treatable structural steel and exploit the rich variety of structures which can be produced by using combinations of deformation and phase transitions and a number of authors have produced ultrafine scale structures by this approach using cold rolling and subsequently tempering low carbon lath martensites [1-5]. It should be noted that cold rolling reduction of 50% i.e. a true strain of 0.8 was sufficient to produce ultra fine grained materials by this route whereas in SPD processing the strain levels are often of the order of 4. Other researchers have used warm rolling and Poorganji et al [6] have used dynamic recrystallisation of an Fe-2%Mn-0.1%C steel to produce ultrafine grain (UFG) ferrite.

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In the current work we have used the concept of combining cold rolling with a subsequent heat treatment but starting with a compositionally graded steel with a C content in the central region of 0.4% which was deformed by cold rolling to a true strain of 1.6. The objective was to use a range of heat treatments at temperatures between 300°C and 900°C to explore the range of fine scale structures which can arise from subsequent heat treatment. The essential objective is that this approach produces not only a refinement in one important length scale e.g. ferrite grain size but a concomitant refinement in related length scales such as carbide size or martensite island size.

Thus the approach yields not only nanoscale structures in bulk materials but provides the opportunity to control both yield strength and competitive processes such as damage initiation and ductility in both ferritic steels containing carbides and in dual phase structure composed of ferrite plus martensite.

2. Experimental methods

The current work was performed on a steel of composition Fe-2.07%Mn-0.4%C-0.4%Cr-0.36%Si-.0.12%Mo. The material was partially decarburised at 1075°C and then water quenched to form a completely martensitic structure at the centre as shown in Fig. 1. It was then rolled using a modified sandwich rolling technique applied to the compositionally graded to produce a highly deformed martensitic which is illustrated in Fig. 2. It should be noted that under these conditions the martensite is completely plastic resulting in a reduction of the lath size as can be seen from a comparison of Fig. 1 and Fig. 2.

![Figure 1. Martensitic structure produced on quenching the compositionally graded steel. Some martensite laths are twinned.](image1)

![Figure 2. Martensitic structure after rolling to a true strain of 1.0. Note the narrow lath size produced by deformation of the martensite.](image2)

This structure was then heat treated over a range of temperatures from 300°C to 900°C. The resulting microstructures were characterized using optical SEM and TEM methods to establish the nature, length scale, and distribution of the various components. Microstructures of as-quenched, cold rolled and heat treated at different temperatures and times were characterized using PHILIPS CM12 TEM, operated at 120 KV. The plane-view TEM foils were prepared as follows. Thin slices were first cut off from samples and ground down (both sides) to a thickness of approximately 80 µm; 3 mm
diameter discs were punched followed by twin-jet electro-polishing in a solution of 10 pct. perchloric acid and 90 pct. methanol, at -35°C and 15V.

The mechanical properties of the structures produced by heat treatment on the rolled martensitic structure were assessed both by hardness measurements and by tensile tests performed at a strain rate of 0.005 sec\(^{-1}\) and the data plotted in the form of true stress–true strain data in the range of uniform strains and the ductility was assessed from measurements of the final reduction in area.

3. Results

The heat treatments of the rolled material in the temperature range 300 to 600°C produced a range of structures with very fine scale ferritic structures an example is shown in Fig. 3. It should be noted that the initial scale of the recrystallised ferritic structure is determined by the scale of the martensite lath size. Thus the recrystallisation process appears to have a very high nucleation frequency and by varying the time and temperature of recrystallisation. It is possible to produce bulk nanoscale structure composed of fine scale spherodised carbides and fine scale ferrite in which the grain size is in the range 10 to 200 nm.

In samples recrystallised at 775°C on quenching from 775°C these showed dual phase structures of the type illustrated in Fig. 4. The scale of the martensitic phase reflects the fine scale of the austenitic phase formed during the intercritical anneal and it is interesting to note that the small scale martensitic phase appears to exhibit a limited number of variants.

The results of the tensile tests performed in the present study are summarised in Fig 5. This shows the true stress true strain curves for the initial martensitic structure formed in the compositional graded structure, the stress strain response after cold rolling to 100% and the stress strain response for the fine scale ferritic/carbide structure and the fine scale dual phase structure.
All the materials are compositionally graded but both the initial as quenched structure and the fine scale dual phase structure exhibit very pronounced elastic-plastic transitions.

![Figure 5](image)

**Figure 5.** True stress-true strain for various treatments applied to a compositionally graded martensitic structure. The fracture strains are based on the reduction in area at fracture. It is of value to note the combinations of strength and ductility and the form of the elastic plastic transition.

4. Discussion

It is clear from the present study that bulk nanostructured steels can be produced even in steels with C levels as high as 0.4%. In the present work we have utilised compositionally graded or architectured materials to achieve high ductility in the original as quenched martensitic structures. The combinations of strength and ductility produced both in the nanoscale ferrite/carbide steels and the fine scale dual phase steels are very attractive and represent an alternative processing route to produce new combinations of properties.

There are many aspects of the nanoscale materials which warrant further detailed study. The recrystallisation on the heavily deformed martensitic structure appears to take place by having a very high nucleation frequency coupled with a restriction of the ferrite grain size to the scale of the initial width of the elongated martensite laths. This is in part due to the simultaneous formation of a fine scale spherodised carbide structure which pins the recrystallised ferrite structure.

In the case of the fine scale dual phase structure this is much finer in scale than dual phase steels produced by conventional intercritical annealing. The simplified martensitic islands which contain only one or two variants are of interest and these also appear to undergo large plastic strain prior to damage initiation resulting in attractive fracture properties. A detailed account of the fracture processes in these nanoscale materials will be reported in a subsequent publication.

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