Effect of cold work and aging on mechanical properties of a copper bearing microalloyed HSLA-100 (GPT) steel

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Abstract. Investigations have been carried out on the effect of cold work and subsequent aging on mechanical properties of a Cu-bearing HSLA-100 steel microalloyed with Nb and Ti. Aging at 400°C after various degrees of cold work (25–70 pct) exhibits multiple hardness peaks. The treatments cause significant improvement in hardness and tensile strength, but at the cost of impact strength. Cold work also causes deterioration in ductility, which again improves on subsequent aging. The C70A treatment involving 70 pct deformation exhibits maximum response to age hardening giving a hardness of 465 VHN and a UTS of 1344 MPa, but with low values of ductility (5 pct) and impact energy (24 J). C50A treatment involving 50 pct cold work and aging results in an optimum combination of mechanical properties. This treatment in the second hardness peak stage yields a hardness of 373 VHN, UTS of 1186 MPa together with a ductility value of 11 pct and impact energy of 109 J. Scanning electron microscopic studies of fracture surfaces reveal that the impact fracture occurs by formation of dimples and nucleation and growth of voids and cracks. Fracture in tensile specimens is caused by formation of voids and cracks at high density striations. Formation of voids and cracks is also assisted by the presence of precipitated carbide particles.

Keywords. Microalloyed steels; striations; peak aging.

1. Introduction

In recent years, various attempts have been made to improve the toughness and ductility of high-strength low-alloy (HSLA) steels by addition of alloying elements (Kim *et al* 2001), special heat treatments (Kim *et al* 1983, 2001; Ingelbriech 1985; Ghosh *et al* 2006; Reip *et al* 2006) as well as by thermomechanical treatments along with suitable modifications in production methods (Furugimi *et al* 1986; Uhashi 1986; Suenaga *et al* 1987; Prasad and Sarma 2005; Bhole *et al* 2006). A need is also felt to develop HSLA steels with very high yield strength together with good toughness and ductility for making many critical components of naval and engineering applications that are subjected to dynamic loading (Czyryca 1993). Due to their age hardening characteristics, Cu-bearing HSLA steels have attracted attention of researchers in the recent past (Skoufari-Themistou *et al* 1999; Dhua *et al* 2001; Ghosh *et al* 2003, 2004; Panwar *et al* 2003, 2005, 2006; Bhagat *et al* 2004; Mao *et al* 2004; Nakada *et al* 2004).

Microalloying of HSLA steels has also received considerable attention (Fernandez *et al* 2006; Reip *et al*

2006; Sugimoto *et al* 2006); these being added to control austenite grain size as well as subsequently to strengthen the transformed structure of ferrite, bainite or martensite. In a recent paper, Panwar *et al* (2006) have reported the influence of Nb, Ti and V on the age hardening characteristics of Cu-bearing HSLA-100 (GPT) steel. It is observed that presence of Ti activates the formation of (Nb, Ti)C precipitates and completely suppresses the precipitation of Cu. These elements also cause multi-stage hardening in the steel. In this paper, mechanical properties developed as a result of strain-induced aging of a microalloyed Cu-bearing HSLA steel have been described. Mechanical properties have been studied at various stages of aging with and without prior cold work.

2. Experimental

The HSLA steel (designated as GPT) investigated in this study was supplied by Naval Research Laboratory, Washington D.C., USA, in the form of a plate of thickness, 51 mm and size, 300 × 200 mm. The chemical composition (wt pct) of the steel was: C 0.05, Mn 1.00, P 0.009, S 0.001, Si 0.61, Cu 1.23, Ni 1.77, Cr 0.61, Mo 0.51, V 0.004, Nb 0.037, Ti 0.003, Al 0.025, Sb 0.003 and As 0.005. Bars of appropriate dimensions were taken from

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the as-received steel plate for various heat treatments. The various treatments were (a) OQ: Solution treatment at 1100°C for 180 min followed by oil quenching, (b) OQA: OQ treatment, followed by aging at 400°C for various times ranging from 5 min to 6000 min, followed by water quenching, (c) C25A: OQ treatment, followed by cold rolling to 25 pct, followed by aging at 400°C for various times ranging from 5 min to 6000 min, (d) C50A: OQ treatment, followed by cold rolling to 50 pct, followed by aging at 400°C for various times ranging from 5 min to 6000 min, and (e) C70A: OQ treatment, followed by cold rolling to 70 pct, followed by aging at 400°C for various times ranging from 5 min to 6000 min.

Hardness (Vickers) measurements were made on test specimens by employing a 30 kg load. Detailed examinations were conducted on oil quenched (OQ), cold worked (CW), peak aged (*P*) conditions in various treatments. The *P* condition referred to the stage of peak observed in hardness-aging curves. Accordingly first, second and third peaks have been designated as *PI*, *PII* and *PIII*, respectively. Tensile tests as per ASTM E8M standard were performed on Hounsfield H25K-S materials testing machine with a crosshead speed of 1 mm/min at room temperature. Charpy V-notch specimens were prepared as per ASTM E23 standard and tested on impact testing machine at room temperature. Although aging process in all the treatments has been studied up to 6000 min, the tensile and impact tests were conducted only up to the *PII* stage. Scanning electron microscopic (SEM) studies were conducted on the fracture surfaces of tensile and impact specimens at 15 kV with LEO 435 VP scanning electron microscope.

3. Results

3.1 Mechanical properties

Figure 1 shows the variation of hardness as a function of aging time. In general, cold working prior to aging increases the hardness, which increases with increasing degree of cold work. Aging at 400°C causes multi stage hardening in all the treatments in this steel. In general, cold working prior to aging reduces the time to reach the first hardness peaks. Accordingly, first peak is observed after 210 min for C25A and after 60 min for C50A and C70A treatments as compared to that after 240 min for OQA treatment. C70A treatment gives maximum response to age hardening thereby giving a hardness value of 465 VHN and a UTS value of 1344 MPa after *PII*. Figure 1 also indicates a complex aging behaviour after *PI* in all the treatments, which is manifested by the different durations of second peaks for various treatments. In complex Cu-bearing steels containing microaddition of Nb and Ti, both of which give precipitation hardening, multiple peaks have been observed at low aging tempera-

tures ($\leq 500^\circ\text{C}$) (Cutler 1979; Krishnadev and Cutler 1979). The as-received GPT steel possesses a UTS value of 685 MPa. Significant improvements in UTS are observed as a result of various treatments. The OQ treatment gives a UTS value of 879 MPa. A substantial increase in UTS is observed after *PI* in OQA and C25A treatments. However, C50A and C70A treatments result in decreased UTS value in *PI* condition. Figure 2 shows that the UTS value generally increases on further aging and attains a maximum value in the *PII* condition. C70A treatment gives maximum UTS value in all the conditions. On the other hand, OQ treatment gives a minimum UTS value of 879 MPa.

The GPT steel in the as-received condition possesses a ductility of 19 pct. The OQ treatment also gives a ductility value of 19 pct. As shown in figure 2, cold working prior to aging causes significant deterioration in ductility. A ductility value of 7 pct is obtained after 25 pct cold work. Cold working to 50 pct causes a marginal increase in ductility to 8 pct. However, further increase in the degree of cold work to 70 pct causes drastic fall in ductility to 5 pct. In general, aging after various degrees of cold work results in increased ductility values. The C25A treatment gives maximum ductility of 16 pct in *PII* condition amongst the cold working and aging treatments. The as-received steel shows an impact value of 341 J. Various treatments cause significant deterioration in impact energy. The OQ treatment gives an impact energy value of 300 J. It is observed that cold working, in general, results in decreased impact energy values. The C70A treatment exhibits lowest impact energy (24 J) in *PII* condition as shown in figure 2. Although the aging process in all the treatments has been studied up to 6000 min, the impact tests were conducted only up to the *PII* stage.

Figure 1. Variation of hardness with aging time.

3.2 Scanning electron microscopy

3.2a Impact specimens: Figure 3 shows the scanning electron micrographs of impact fracture surfaces after various treatments. The SEM study after various stages shows that on impact loading the fracture occurs by the

Figure 2. Mechanical properties of GPT steel in various conditions.

formation of dimples, nucleation and growth of voids at the precipitate/inclusion sites, and development and propagation of cracks. The high impact energy values are characterized by fine network of ductile dimples and closely spaced coarse voids, which are linked by several finer voids, whereas low impact energy values are characterized by (i) shear dimple networks, (ii) coarse voids, (iii) quasi cleavage facets, (iv) intergranular cracks and (v) stepped fracture appearance.

High impact energy observed in OQ condition (300 J) is characterized by fine network of ductile dimples and closely spaced fine voids (figure 3a), which is indicative of a high resistance to cleavage fracture. Deformation by cold rolling results in drastic fall in impact strength. The fracture in C25A treatment is characterized predominantly by quasi cleavage facets, which is in accordance with a poor impact value of 73 J and hence poor resistance to cleavage fracture. On the other hand, increase in impact value (132 J) after 50 pct cold work is characterized by fine network of shear dimples and closely spaced voids. Further reduction to 70 pct exhibits a quasi cleavage appearance together with development of cracks (figure 3b) conforming to a low value of impact energy (77 J) observed in CW condition of C70A treatment.

Aging, in general, reduces the impact strength in various treatments. The drastic fall in impact value (203 J) in P condition of OQA treatment is characterized by a relatively coarse network of ductile dimples and coarse voids. The poor impact energy values in PII condition of C25A (39 J) and C70A (24 J) are characterized predominantly by quasi cleavage appearance and intergranular cracks (figures 3c, d). However, the relatively higher value of impact energy (109 J) in PII condition of C50A treatment is manifested by mixed appearance of quasi cleavage and shear dimples. It is observed that presence of precipitate particles generally helps in nucleation of voids in impact tests.

3.2b Tensile specimens: The drastic fall in ductility after various degrees of cold work is accompanied by coarsening and shearing of dimples and voids together with the development of cracks in the central region, and coarsening of striations in the peripheral zone. Accordingly, the drastic fall in ductility after 25 pct cold work (7 pct) is characterized by a network of coarse shear dimples and coarse voids (figure 4a). Figure 4(b) shows the scanning electron micrograph in CW condition of C50A treatment, which shows networks of coarse striations and fine cracks. Such structural features are responsible for poor resistance to tensile fracture and hence a poor ductility value (8 pct). Heavy deformation to 70 pct cold work causes further decrease in ductility (5 pct). This is reflected in SEM study by the uniformly distributed coarse voids together with coarse and fine cracks.

Aging after cold work, in general, increases the ductility. Accordingly, significant improvements in ductility are

Figure 3. Scanning electron micrographs of impact fracture surfaces showing (a) fine network of ductile dimples and closely spaced fine voids in OQ condition (300 J), (b) quasi-cleavage appearance and nucleation of cracks after 70 pct cold work (77 J), (c) quasi-cleavage appearance and shear dimples in PII condition of C25A treatment (39 J) and (d) intergranular cracks in PII condition of C70A treatment (24 J).

observed in *P* conditions. High ductility value of 16 pct in *PII* condition of C25A treatment is characterized by fine network of shear dimples, extremely fine voids and fine cracks (figure 4c). The ductility in *PII* condition decreases with increasing degree of cold work. The poor ductility (11 pct) in C50A treatment is manifested by fine network of shear dimples, coarsening of voids and cracks. The scanning electron micrographs corresponding to extremely poor ductility value (8 pct) in C70A treatment exhibit predominantly quasi cleavage appearance together with coarse and straight striations with fine cracks in the peripheral zone (figure 4d).

4. Discussion

The mechanical properties in GPT steel are significantly influenced by the processing conditions and are closely

linked with the degree of cold work and the aging behaviour. In a recent study, Panwar *et al* (2006) reported in detail the aging characteristics of this steel. As shown in figure 2, aging allows increase in hardness and UTS, but at the cost of impact energy. Aging after cold work poses a complex situation for explanation of mechanical properties. Figure 2 shows that hardness increases with increasing degree of cold work and aging time. On the other hand, there is continuous increase in UTS in OQA and C25A treatments. In C50A and C70A treatments, the UTS first decreases slightly up to *PI* stage, and then increases on further aging to *PII*. It is also observed that UTS increases with increasing degree of cold work. Accordingly, C70A treatment gives maximum UTS values in all the conditions. The resultant increase in hardness and UTS is attributed to the coarsening, and fresh nucleation and growth of fine (Nb, Ti)C carbide particles in the

Figure 4. Scanning electron micrographs of tensile fracture surfaces showing: (a) network of coarse shear dimples together with coarse voids after 25 pct cold work, (b) network of coarse striations after 50 pct cold work, (c) fine network of shear dimples, extremely fine voids, fine cracks in PII condition of C25A, and (d) quasi-cleavage appearance and coarse and straight striations with fine crack in PII condition of C70A.

matrix. The initial drop in UTS in PI condition of C50A and C70A treatments is due to the coarsening of (Nb, Ti)C carbide precipitates. On the other hand, increase in hardness and UTS on aging after various degrees of cold work is attributed to the enhancement of dislocation density developed due to cold work, which subsequently forms tangles with pre-existing (Nb, Ti)C carbide precipitates (Panwar *et al* 2006). Hence, the strong pinning effects of dislocations and (Nb, Ti)C carbides provide sufficient dislocation-precipitate strengthening in this steel.

Cold working prior to aging, in general, results in significant deterioration in ductility, which is a matter of concern and may adversely affect the use of this steel in thermomechanically aged conditions. Bakaloglu (2002) also observed that the ductility in HSLA steels decreases with increase in the rolling reduction up to 40 pct. Aging

after cold working improves ductility in all treatments. The primary factor responsible for poor ductility is the degree of work hardening. In addition, ductility to some extent also appears to be adversely affected by the presence of coherent precipitate particles. Accordingly, it is seen that in PI condition in OQA treatment there is some drop in ductility primarily due to the presence of coherent (Nb, Ti)C precipitates (Panwar *et al* 2006). Coherent precipitates cause inhomogeneous deformation and early crack nucleation. The stress concentration in the vicinity of coherent precipitates is also high, which leads to easy crack nucleation and propagation resulting in poor ductility. The scanning electron micrographs (figure 4) also show existence of ductile dimples together with wavy striations in OQ condition, reflecting good ductility. In the PI condition the striations become less wavy (straight)

and due to high stress concentration at the junction of these striations cracks are nucleated very early leading to failure and loss of ductility.

Figure 5. Variation of impact energy with (a) hardness, (b) UTS and (c) ductility.

In the CW condition lowest ductility is observed in C70A treatment owing to very high level of work hardening after 70 pct cold work. On aging after cold work, there is slight polygonization of the matrix (Panwar *et al* 2006), leading to some improvement in ductility. Although aging after cold work causes formation of coherent (Nb, Ti)C carbide precipitates which are deleterious for ductility, the substantial improvement in ductility due to polygonization overcomes the deleterious effect of coherent precipitates. The rate of polygonization and recrystallization is slowest in C70A treatment due to extremely high dislocation pinning effect, which yields only small improvement in ductility on aging to PI and PII stages. On the other hand, as revealed in figure 2, improvement in ductility in C25A treatment on aging to PI and PII conditions is substantial. In this treatment, the annihilation of dislocations and the process of polygonization are rapid, which result in a good ductility (16 pct) in the PII condition. The impact strength suffers a serious setback as a result of various treatments studied in this investigation (figure 2). The impact energy is very sensitive to the presence of coherent precipitates and the dislocation network in the matrix. It is now well established that presence of coherent precipitates causes inhomogeneous deformation leading to crack nucleation. Coarse precipitates are not as much dangerous to impact strength as the fine coherent precipitates, since the stress concentration around coarse precipitates is always much lower than that in the vicinity of coherent precipitates. At the same time coarse precipitates do not result in inhomogeneous deformation. Therefore, in all treatments substantially low impact energy values are obtained on aging to PI and PII stages. As regards the influence of cold work, the impact energy is predominantly related to the intensity of stress concentration in the matrix. Low deformation, as in C25A, causes accumulation of dislocations mainly at grain boundaries (Panwar *et al* 2006). Thus grain boundaries become centres of very high stress concentration, which results in poor impact energy (73 J). In 50 pct CW condition, the grain boundaries are almost eliminated and the average dislocation density of the matrix becomes lower than that at grain boundaries in 25 pct CW state. Thus the lower stress concentration in the matrix results in higher impact energy (132 J). Very severe cold work, as in C70A, again raises stress concentration in the matrix, which causes rapid nucleation of cracks leading to drastic lowering of impact energy (77 J). Thus, it is seen that in C50A treatment the overall impact energy level is higher than that in C25A and C70A treatments. Aging after cold work influences impact energy depending upon the density of formation of coherent (Nb, Ti)C precipitates as also the dislocation density as affected by the aging process. In the C70A treatment, continuous and steep decline in impact strength is due to slow recrystallization and a high density of dislocations remaining even up to the end of the aging process, as is also revealed by the TEM obser-

variations reported in an earlier paper (Panwar *et al* 2006). With increase in degree of cold work there is also an increase in the density of fine/coherent precipitates, which also contributes to lowering of impact energy. Due to partial recrystallization and lower density of coherent precipitates in C25A and C50A treatments, there is slight improvement in impact energy up to PI condition. However, due to increased concentration of fine and coarse precipitates the impact energy in these treatments decline substantially in PII stage of aging as seen in figure 2.

Figure 5 shows the variation of impact energy as function of (a) hardness, (b) UTS and (c) ductility. In general, the impact toughness decreases with increase in hardness and UTS, whereas its correlation with ductility shows a general increase with increasing ductility. The large scatter in the data predicts that no single parameter is solely responsible for any increase or decrease in toughness; it also depends on other parameters such as microstructure, distribution of precipitates and dislocation density. SEM study (figures 3 and 4) shows that high ductility and impact values are associated with fine network of ductile dimples and high density of closely spaced voids. The tensile specimens also exhibit high ductility by existence of network of fine and wavy striations in the peripheral zone. Maropoulos and Ridley (2004) reported that the adjacent voids are usually linked by several finer voids. Hausild and Berdin (2002) proposed that these finer voids are nucleated presumably by fine carbides or very small inclusions as a consequence of the high local strains required to separate the intervening matrix. Poor ductility and impact values are accompanied by predominantly quasi-cleavage facets/patterns, coarse network of shear dimples, coarse voids and development of cracks. The SEM study of peripheral zone of tensile fracture surfaces shows network of coarse and straight striations corresponding to low ductility. According to Maropoulos and Ridley (2004), the shear dimples are mainly observed at the shear lip zone of charpy and tensile specimens. These regions display a well defined network of oval dimples elongated in the direction of shear. With shear dimples it is not always possible to identify whether the crack initiating particle is an inclusion or carbide because it may be hidden below the surface.

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