DEVELOPMENT OF EUTECTOID STEEL FOR WIRE-ROPE APPLICATION POSSESSING ENHANCED STRENGTH AND WEAR RESISTANCE

A Thesis Submitted

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Synopsis

In the quest of world-wide efforts on new material development, the broad objective of the present research work is set as the development of a cost-effective material for high-strength wear-resistant structural application, through a novel synthesis route of high-voltage low-current electric energy input, that would possess: (i) reasonable hardness (above 400 HV), (ii) ultra-high strength (UTS above 1 GPa), (iii) significant ductility (above 15% Elongation) and (iv) superior wear resistance with respect to the conventionally synthesized material.

Indeed, the distinctive synthesis approach of high-voltage low-current (HVLC) electric energy input is adopted for the first time in the present research work; where current (150 mA), so the current density (1.19 Acm^{-2}), is kept low to negate Joule heating effect. On the other hand, the applied voltage is kept at high level (25-100 kV) so as to augment the electromigration process. Such a typical voltage-current combination is made feasible by designing a typical electrical circuit at our high voltage laboratory of National Institute of Technology Durgapur, India. In particular, the presence of porcelain insulator in this electrical circuit ensures to maintain a low current along with a 'high voltage electric stress' that would result in atomic displacement in the specimen. In this regard, the applied voltage range (25-100 kV) is so chosen that the resultant electric energy input well exceeds the cohesive energy of iron so as to cause atomic migration.

The plain carbon eutectoid steel (AISI 1080 steel, a structural material) is chosen as the study material due to its low cost (in absence of costly alloying elements) and an inherent constitution of a typical mixture of a hard phase (cementite) and a soft ductile phase (α-ferrite); thereby keeping in mind the retention of adequate ductility while enhancing hardness, strength and wear resistance. Furthermore, AISI 1080 steel is often used as wire-ropes in mine haulage, mooring, bridge, cranes, lifts etc. where the resistance against tribological surface damage (wear) becomes critical. This further necessitates to investigate temperature dependent wear damage characteristics of AISI 1080 steel (not studied hitherto) in accordance to its application where, in reality, the mine temperature could rise up to 60°C. Since atoms at surface are more mobile than the interior (bulk), in presence of very hard abrasive counter material and considerable applied load, a significant tribological surface damage is quite expected even for a temperature rise up to 60°C. Indeed, in view of the conceived philosophy behind high-

voltage low-current electric energy input, the augmented electromigration process would cause nano-scale structural refinement and matrix supersaturation so as to greatly enhance, hardness, strength, wear resistance along with restoration of reasonable ductility. The novelty of this research work is exemplified with the conceptual leap forward pertaining to the adoption of a new synthesis route, cost-effective material development and novel microstructure evolution at nano-scale to provide ultra-high strength coupled with large ductility and wear resistance.

In pursuance of meeting the objective, consequent to high-voltage low-current treatment, the microstructure evolution is investigated by Optical Metallography, Field Emission Scanning Electron Microscopy (FESEM), X-ray Diffraction (XRD) analysis, Atom Probe Tomography and High Resolution Transmission Electron Microscopy (HRTEM) along with Selected Area Electron Diffraction analysis. Standard hardness and tensile tests are carried out to ascertain the hardness, strength and ductility properties. In order to study the failure mode in correlation with microstructure and properties, the tensile fractured surfaces are studied in FESEM. Furthermore, temperature dependent dry sliding wear behaviour of the selected specimens of prime importance is investigated in a pin-on-disk wear testing machine against an alumina disk. Following wear test, worn surfaces are characterized with FESEM along with FESEM based EDS analysis, XRD analysis and Raman Spectroscopy. Besides, wear debris are characterized with FESEM along with FESEM based EDS analysis.

The microstructures of both as-patented (specimen code: PT) and patented followed by cold drawn (specimen code: CD) AISI 1080 steel specimens reveal a lamellar phase mixture of α-ferrite and cementite, known as pearlite. Quantitative estimation in view of FESEM micrographs reveals that the cold drawn specimen has a reduced interlamellar spacing (86±8 nm) than that in as-patented condition (118±10 nm). When this patented-cum-cold drawn AISI 1080 steel, initially possessing a lamellar pearlitic structure, is treated with high-voltage (25-100 kV) low-current (150 mA) energy input (above the cohesive energy) a unique modification of microstructure is realized in view of morphology of microconstituents, structural refinement and matrix supersaturation. On a gross scale, four distinctive regions are identified; which are: lamellar pearlite often containing nucleated cementite spheroids (Region-I), fragmented cementite lamella in α-ferrite matrix (Region-II), submicroscopic cementite particles/clusters dispersed in α-

ferrite matrix (Region-III), and supersaturated α-ferrite (Region-IV). Such a significant structural alteration is definitely an effect of fine-scale atomic migration under high voltage low current (HVLC) treatment; where the energy input well exceeds the cohesive energy of the base metal. Indeed, the initial microstructure of steel in cold drawn condition is expected to have variations in chemical potential at different locations of lamellar pearlite. Prior to HVLC treatment, the defect-enriched lamellar fault sites are readily identified in cementite lamella. The migration of atoms from lamellar fault site (the region of higher chemical potential) towards adjacent matrix would result in fragmentation of cementite lamella into different shapes. Depending on local deviation from equilibria (in turn the local chemical potential), the process of lamellar fragmentation of cementite may be at an initiation stage (originating Region-II) or more intense (originating Region-III). Moreover, the nucleation of cementite spheroids (as in Region-I) is a consequence of atomic migration from the lamellar fault site to the adjacent flat surface within same lamella. Furthermore, atomic migration from the surface of the cementite lamella into α-ferrite matrix in a uniform manner (in other words, uniform dissolution) would generate supersaturated α-ferrite solid solution (Region-IV).

Simultaneous effects of matrix supersaturation (generating martensite in Region-IV) and structural refinement are observed to progress up to a treatment duration of 5 minutes at a particular applied voltage. This trend reverses henceforth till 10 minutes of treatment duration, resulting in structural coarsening and elimination of matrix supersaturation. On the other hand, the overall effect of applied voltage may be summarized as the peak-broadening of martensite and structural refinement with increasing voltage up to 100 kV. Most importantly, for an optimum duration (5 min) of high-voltage low-current treatment at 100 kV, a unique microstructure evolves that consists of dispersion of nano-sized hard cementite spheroids (mean Size = 23±9 nm) and clusters (mean size = 43±7 nm) in nano-thick martensite crystals (mean size = 41±10 nm) of stratified tile-morphology together with distributed α-ferrite regions containing sub-microscopic cementite particles of various shapes. The high-voltage low-current induced martensitic transformation is observed for the first time. Moreover, the typical substructural features include a high dislocation density (of the order of 10^{15} m^{-2}) in the martensitic regions and systematically

arranged dislocations of similar sign at incoherent cementite particle-martensite matrix interface.

The structural modifications achieved eventually result in a significant improvement in mechanical properties. At a particular voltage, the bulk hardness and strength increase with treatment duration till 5 min and thereafter decreases. On the other hand, at a particular treatment duration, bulk hardness and strength increase with increasing applied voltage. At any combination of voltage and treatment duration, with regard to microhardness test results, Region-IV possesses the maximum hardness, immediately followed by Region-III. This is primarily attributed to martensitic strengthening effect in Region-IV (due to the presence of supersaturated α-ferrite/martensite phase) and dispersion strengthening effect in Region-III (due to dispersed individual cementite particles and clusters of cementite particles of submicroscopic size in α-ferrite matrix). In addition, for a treatment duration of 5 min at higher voltages (75 kV and 100 kV), unlike lower voltages (25 kV and 50 kV), the Region-IV contains an additional feature of nano-sized cementite particles dispersed in martensite matrix so as to provide further effect of dispersion strengthening along with martensitic strengthening in Region-IV, thereby providing maximum hardening/strengthening effect.

Therefore, at a particular voltage, with gradual matrix supersaturation (thereby augmenting martensitic strengthening effect) and structural refinement (thereby promoting dispersion strengthening effect) up to 5 min treatment duration, microhardness of these regions; and hence the bulk hardness, is gradually enhanced and thereafter falls due to the reversal of these effects. Again, by virtue of increased internal strain in martensitic structure (as reflected in increased full width at half maxima), the hardness value of Region-IV increases with increasing voltage. Again, the cementite particle as well as cementite cluster sizes are found to decrease with increasing voltage, resulting in enhanced dispersion strengthening effect in Region-III. Accordingly, as an overall effect, bulk hardness and strength increase with applied voltage.

With regard to the conjoint effect of process parameters enumerated above, the highest hardness (448 HV) and strength (UTS = 1461 MPa) are achieved at 100 kV with a treatment duration of 5 min due to maximum feasible combination of martensitic strengthening and dispersion strengthening effects. Although hardness and strength are enhanced to a high level, significant ductility is still retained (%Elongation = 20 at 100

kV, 5 min) due to the dimple rupture mode of failure augmented within soft α-ferrite phase and at the interface between dispersed submicroscopic spheroidal cementite particle and α-ferrite matrix.

Indeed, negating the mutually exclusive strength-ductility relationship, a new pathway is hereby invented in material development for low-cost structural application so as to achieve ultra-high strength (UTS ~ 1.5 GPa), significantly high specific strength (188 MPa/g cm^{-3}) and large ductility (%Elongation = 20) through high-voltage low current treatment of cold drawn AISI 1080 steel with 5 min hold at 100 kV.

Finally, the temperature dependent wear behaviour of investigated AISI 1080 steel, in general, exhibits several aspects of characteristic wear mechanism; namely, adhesion, microcutting abrasion, microploughing abrasion, plastic flow and tribo-oxide formation depending on prior treatment and test temperature. The AISI 1080 steel, synthesized through conventional treatment of patenting followed by cold drawing, suffers from aggravated wear loss with increasing test temperature. Although the evolution of an adherent iron oxide layer at surface provides some wear resistance at room temperature (30°C); the breakdown of this layer at higher test temperatures and dominating thermal softening effect (readily negating strain hardening effect) leading to severe microcutting and microploughing abrasive wear coupled with plastic flow raise the overall wear rate to a high level, in particular, at 60°C. On contrary, the newly developed high-voltage low-current electric energy treated AISI 1080 steel (treatment condition: 100 kV, 5 min), although initially suffers from enhanced wear loss above room temperature, the overall wear rate again falls down at 60°C, corresponding to the highest mine temperature of real service situation. In case of high-voltage low-current electric energy treated steel, nano-void nucleation is often found to inhibit surface crack propagation to reduce overall wear rate. Although the adherent oxide layer, initially developed at room temperature (30°C), appears to be dismantled at intermediate test temperature regime (40°C and 50°C) leading to an accentuated microcutting and microploughing abrasive wear; a typical adherent tribo-oxide layer of $FeAl_2O_4$ is developed at 60°C. The adherent tribo-oxide layer eventually lowers down the surface friction (and so the surface temperature), thereby negating thermal softening effect to arrest the wear loss at 60°C. This declares an emergence of a wear resistant high-voltage low-current electric energy treated AISI 1080 steel to be applicable even at the highest mine temperature of 60°C.

With regard to main techno-scientific achievement, understanding the necessity of high strength ductile wear-resistant material development in view of more interphase-interface creation, distribution of submicroscopic hard phase/phases in a soft ductile matrix, a new synthesis route of high-voltage low-current (HVLC) energy input is applied on conventionally treated (patented-cum-wire drawn) AISI 1080 steel (inherently possessing and combination of hard and soft phases with lamellar morphology). In accordance, for an optimum duration (5 min) of HVLC treatment at 100 kV a unique microstructure evolves that consists of dispersion of nano-sized hard cementite spheroids and clusters in nano-thick martensite crystals of stratified-tile-morphology together with distributed α-ferrite regions containing sub-microscopic cementite particles of various shapes. Such a novel microstructure provides ultra-high strength (UTS ~ 1.5 GPa), significantly high specific strength (188 MPa/g cm^{-3}) and large ductility (%Elongation = 20) and significant wear resistance so as to pave a new pathway is in material development for low-cost structural application.