NIOBIUM IN MICROALLOYED ENGINEERING STEELS, WIRE RODS AND CASE CARBURIZED PRODUCTS

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Abstract

Microalloying technology, developed for the production of flat products (plate, strip and line pipe) during the 1960’s and 1970’s, has been applied to “long products” such as engineering bars, sections, forgings and wire rod since about 1980. In the 1980’s the main rationale to use niobium bearing steel bars and wires was to eliminate the need for a hardening process, i.e. quenching and tempering, in manufacturing heat-treated steel parts without any trade off in properties. Owing to the significant cost reductions and energy savings by adopting microalloyed steels, they have replaced conventionally heat-treated steels for connecting rods, suspension components and fasteners etc., in the automotive industry. On the other hand, the effect of Nb on the mechanical properties of heat-treated parts has also been clarified, and new steel grades, e.g. high strength steels for fasteners with 1500 MPa tensile strength and spring steels used in 1200 MPa design stress applications, have been developed. Furthermore, the immense capacity of niobium precipitates in steel to prevent grain coarsening during heat treatment has been utilized in developing case hardening steels for transmission gears and CVJ shafts, which are cold-forged and then carburized at a high temperature. The new grades have also succeeded in eliminating softening treatments such as spheroidized annealing and/or bright normalizing treatment in the manufacturing sequence.

Introduction

As microalloying enables high strength parts to be produced in the as-forged condition without a subsequent cost consuming reheat, quenching and tempering, the application of microalloyed steels has been extended. Even though Nb is obviously a dominant element for microalloying as-processed steels, it is now also being used to improve the properties in heat-treated steels.

Within the last twenty years the strength level of structural steel parts used in automobiles has conspicuously increased in line with strong demands from the automotive industry. Figure 1 summarizes the change in fatigue limits and toughness of heat-treated steels for fasteners, springs and case hardened parts, respectively. The achievement of improving mechanical properties in each grade, without raising total manufacturing costs, is attributed not only to improved alloy designs but also to process innovations, i.e. hard shot peening technology applied to carburized gears and hot setting and warm peening for springs.

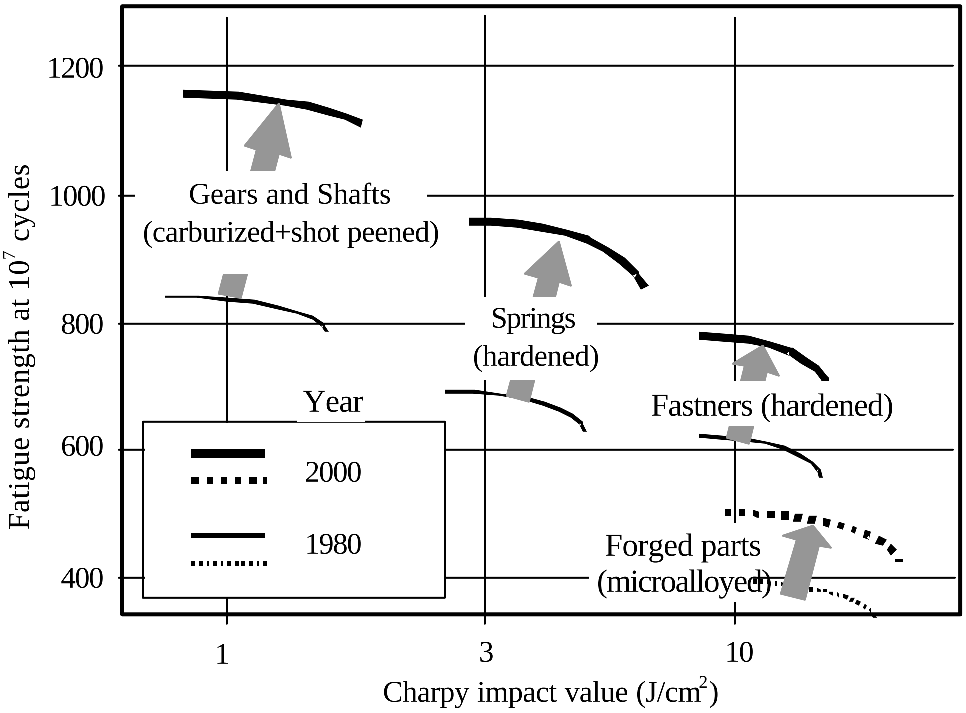

Figure 1: Change in the upper limits of fatigue strengths and toughness balance in structural parts during the past 20 years.

The emphasis should be on niobium additions to heat-treated grades, which would be associated with the prevention of grain coarsening during a heat treatment with resultant brittleness and coupled with an increase in the strength level of manufactured components.

This paper reviews practical examples of Nb additions to engineering steels for forged parts, fasteners and springs. In carburizing steels, the process and heat treatment are so complicated that there are difficulties to apply Nb to this grade. Therefore, research results concerning a developed steel for carburized gears and shafts is described in detail: the niobium-bearing steel has superior cold formability without spheroidizing annealing after rolling and high resistance to grain growth during carburizing even when it is not normalized after cold forming.

Forging Steels

Niobium is viewed as the key ingredient in many high-strength, low-alloy steels (HSLA) and regarded as the most effective grain refiner and a promising element for precipitation hardening in hot-rolled products (1). This favorable effect of Nb was soon applied to hot forging steels at the beginning of the 1970's. Hulka et al. (2) reviewed the historical development of the forged steels containing Nb and physical metallurgy of Nb in medium carbon steels, which are the most popular forging bar steels.

Hot-forged Products

For closed die forgings in the automotive industry the steel grade 49MnVS3 (0.50% C-0.25% Si- 0.70% Mn-0.040% S- 0.10% V) was established in the middle of the 1970's (3). Though the required strength could be obtained by 0.08% Nb microalloying when high processing temperatures are applied, the limited toughness restricted its application. For improved toughness, a 0.38% C-1.00% Mn-0.07% V-0.03% Nb steel was developed and applied in the as-forged condition for automobile parts with improved safety (4).

Bucher (5) compared the structures of AISI 1141 – niobium-modified steel with that of standard AISI 1141 steel taken from forged connecting rods. The structural refinement increased the hardness of the niobium bearing steel to 97 HRB vs. 92 HRB for the standard grade, and improved toughness that was adequate for connecting rods. The AISI 1141 - niobium-modified steel is also used for weld yoke or universal joint couplings (6).

A French steel company developed a variety of microalloyed forging steels including high strength steel with tensile strength levels over 1000 MPa. The basic compositions of their grades is 0.45% C-0.30% Si-1.50% Mn-0.12% V-0.04% Nb, and a constant ultimate tensile strength is obtained by way of a compromise between bainite formation and vanadium precipitation during air-cooling. Niobium's roll is in controlling the austenitic grain size and, hence, keeps the steel hardenability stable. These grades are used for suspension arms and anti-sway bars in the automotive industry.

Future Prospective of Controlled Forging Steels

Sampei et al. (7) confirmed that Nb-added medium carbon steel bars (0.25 – 0.45% C) had a good strength - toughness ratio after controlled rolling, where a billet of 114 mm^2 was heated to 1373 K and then rolled in to 22 mm round bars with a finishing temperature of 1073 K. This intermediate finishing temperature was adequate to suppress recrystallization of austenite in niobium-bearing steel bars and resulted in grain refinement, leading to high mechanical properties.

If this controlled rolling technology is applied to the forging process, it is anticipated that superior mechanical properties in forged parts can be obtained. Hulka (2) referred to the idea of controlled forging, which had been conceptualized by Pawelski et al. in 1975 (8), and demonstrated that finishing forging temperature in the range 950 – 1100 K allowed for a better balance of tensile strength and toughness in niobium-bearing steels (0.32% C, 1.47% Mn) than in conventional steel. Predictably, a high load during forging at an intermediate temperature shortened the life of the forming dies. This poor productivity limits the use of controlled forging.

Recent studies on grain refining technology has revealed that the mechanical properties are dramatically improved when the grain size becomes finer, to less than a micron (9). Because finer grains can be easily attained with a niobium-bearing steel deformed at a warm forging temperature, the controlled forging of niobium steel would be effective for practical use in the future.

Steels for Fasteners

Steel wire rods for high strength fasteners, in which the tensile strength is over 800 MPa, are required to have cold formability, good mechanical properties (tensile strength, proof stress, fatigue strength and toughness) and resistance to delayed fracture when the tensile strength reaches up to 1200 MPa. The key stages of development in steels for high strength fasteners can be summarized; the adoption of microalloyed wire rods to non heat-treated fasteners, the reduction in the amount of alloying element from heat-treated grades and the prevention of delayed fracture in high strength fasteners.

Adoption of Microalloyed Wire Rods to Non Heat-treated Bolts

In 1980, Gondo et al. (10) developed a steel wire for cold-headed bolts which exhibited a strength level of 700 MPa without spheroidized annealing, hardening or tempering. The steel composition was 0.10% C-1.5%Mn along with a small amount of Nb, V and Ti, suitable for obtaining moderate ferrite and pearlite microstructures under controlled rolling and cooling conditions.

Heritier et al. (11) reported that the fabrication of class 8.8 bolts from 0.2% C-1.2 % Mn -20 to 50 ppm B grade is an industrial reality. With a high hardenability in steel due to a boron addition the low carbon bainite was initiated during cooling from the rolling temperature. They found that the low carbon bainitic ferrite had a high strain hardening exponent and demonstrated that the combination of 0.12% C-1.65% Mn- 0.08% Nb-25 to 50 ppm B and the high ratio of wire drawing could produce a wire rod with a strength level range of 1000 – 1200 MPa. Niobium is added to strengthen the steel via grain refinement and interphase precipitation.

A ferrite-pearlite steel with a small amount of precipitation and grain refining elements has been designed to meet the specification of class 8.8 fasteners (12). Table I shows the chemical compositions of the steel. In processing, low temperature soaking and finish rolling at a low temperature were adopted to obtain the extremely fine-grained steel and, after rolling, controlled cooling was carried out to promote precipitation hardening. The effect of the cooling rate on the hardness of hot worked specimens is shown in Figure 2. The hardness increment compared with the base steel, 0.14% C-0.25% Si-1.45% Mn-0.1% Cr, is 30-45 HV in the developed steel which stems from the combined effects of initial grain refinement and precipitation hardening by vanadium and niobium carbonitrides.

Table I Chemical compositions of test steels (wt %)

Grade	C	Si	Mn	P	S	Cr	V	Nb	sol.-Al	N
Base Steel	0.14	0.26	1.43	0.011	0.003	0.10	-	-	-	0.001
V	0.14	0.25	1.44	0.010	0.003	0.11	0.10	-	-	0.001
Al-N	0.14	0.25	1.46	0.011	0.003	0.10	-	-	0.033	0.012
Nb	0.14	0.26	1.44	0.010	0.003	0.10	-	0.02	-	0.001
V-Nb-Al-N	0.14	0.24	1.47	0.010	0.003	0.10	0.10	0.02	0.032	0.013

The increment in hardness in steel wire sometimes deteriorates the life span of dies and can mitigate the cost saving derived from eliminating a hardening-tempering process. An aging process may prove promising in strengthening relatively low strength cold-formed fasteners. Boratto et al. (13) designed a niobium plus titanium microalloyed steel, 0.09% C-0.2% Si-1.9% Mn-0.6% Cr-0.04% Nb-0.03% Ti, and produced a wire rod with an acicular ferrite structure having an as-rolled tensile strength around 900 MPa. They confirmed that a low temperature aging at 523 K increased both the strength and the ductility of the cold-headed fasteners to meet all specifications for strength class 10.9.

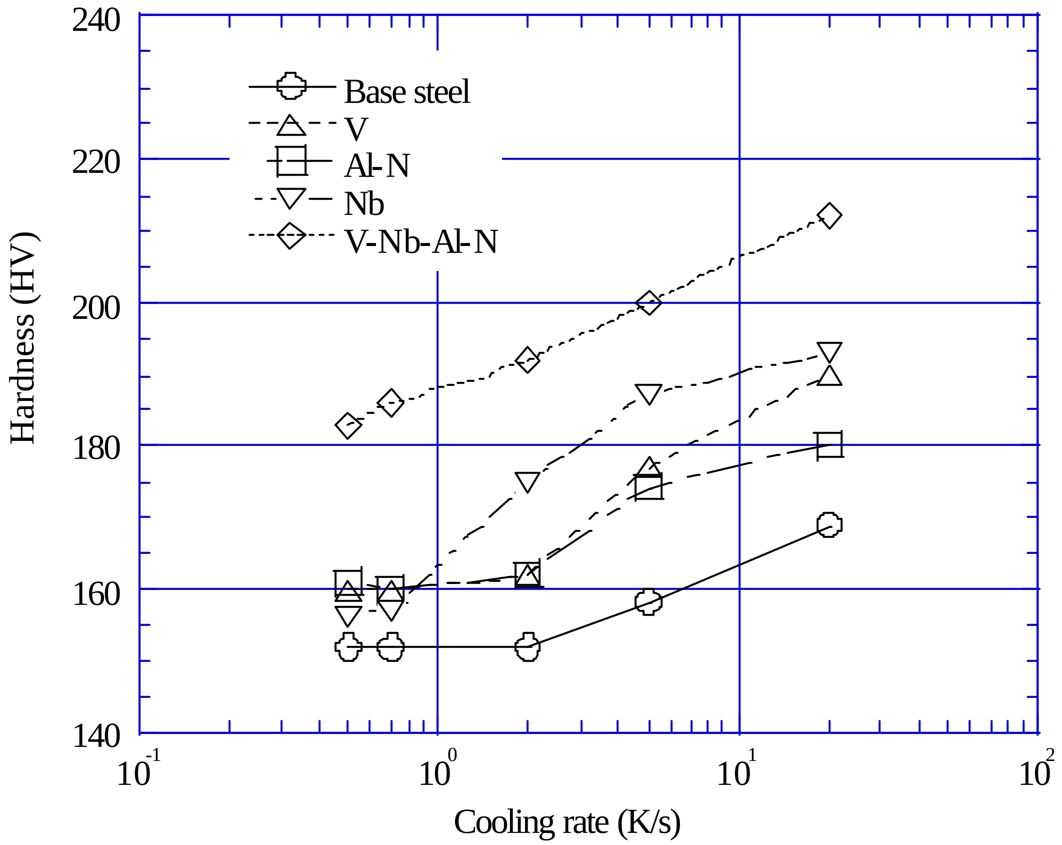

Figure 2: Effect of cooling rate on the hardness of hot worked specimens.

Application of Nb to Hardened-Tempered Steel for High Strength Fasteners

Generally, Class 10.9 fasteners are manufactured using chromium-molybdenum steels like JIS SCM435, equivalent to SAE 4137, through quenching and tempering after cold heading. Since the hardness of this grade after wire rolling is too high for the fasteners to be drawn and cold headed, annealing treatment must be conducted so as to lower the hardness in the rolled wire.

A new grade, 0.25% C-0.05% Si –1.0% Mn-0.3% Cr-0.05% Ti-0.025% Nb-20ppm B steel, was developed and delivered in a controlled rolled condition and cold headed without softening treatment (14). The deformation resistance of this grade even in the as-rolled state is low enough to be cold formed due to reduced levels of C, Si and Cr, which strengthen the steel matrix. Boron and niobium are added to compensate for the lack of hardenability and to obtain grain refinement, respectively.

When tensile strength in fasteners exceeds 1200 MPa, common quenched-tempered alloy steels are vulnerable to be attacked by hydrogen in a service environment and this could result in delayed fracture. In order to reduce susceptibility to delayed fracture, it is suggested that impurities be reduced at the prior-austenite grain boundaries, plus austenite grain refinement, as well as a change in carbide precipitate morphology at the boundaries. A delayed fracture resistant steel with 1500 MPa tensile strength is proposed having a steel chemistry: 0.35% C-0.20% Si-0.35% Mn-0.010% P-0.010% S-1.25% Cr-0.40% Mo-0.02% Nb. A modification in steel chemistry, i.e., reducing P, S and Mn and increasing Cr and Mo together with Nb additions to JIS-SCM435 (0.35% C-0.85% Mn-0.020% P-0.015% S-1.0% Cr-0.20% Mo) has been proved to markedly improve delayed fracture resistance (15).

Spring Steels

High strength quenched-tempered spring steels are required to have desirable tensile strength, proof stress, fatigue strength, sag resistance, and, with increase in the design stress, corrosion fatigue strength and delayed fracture resistance. Figure 3 shows the change in the design stress of coil springs in the Japanese automotive industries. In the early 1990's, light springs were necessary for reducing weight in passenger cars and high-alloy steels were developed to endure a surge in design stress accompanied by a thinner coil or plate shape. Recent developments in this field have drifted back towards reducing alloying elements in spring steel, more equivalent to 1200 MPa design stress spring.

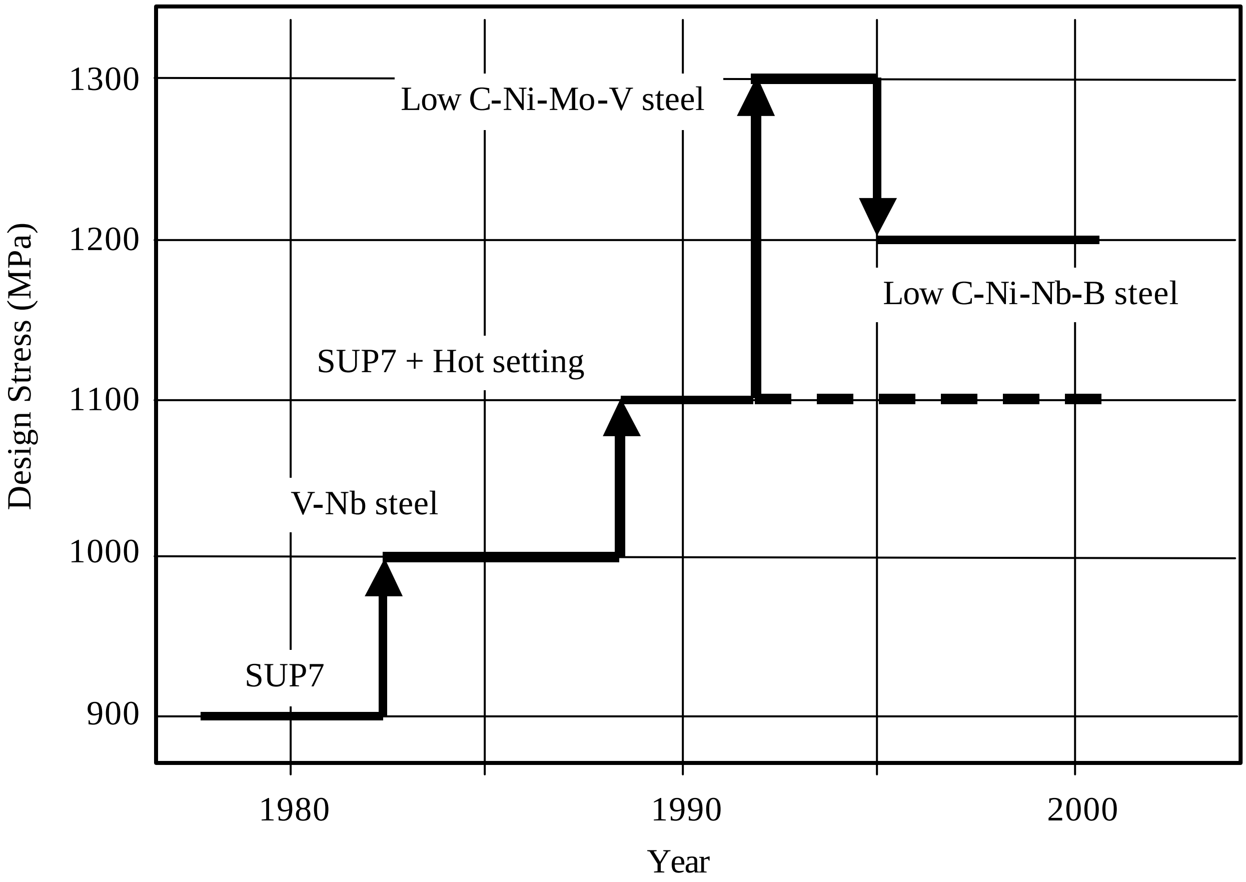

Figure 3: Change in the design stress of commonly used hardened coil springs.

Coil Spring Steels

In 1981 an attempt (16) was made to improve sag resistance in coil springs by additions of V and Nb to JIS SUP7, equivalent to SAE 9260. Superior sag resistance in vanadium and niobium-bearing steel than that of SUP7 was observed and attributed to the combination of a solid solution effect by V, precipitation hardening due to vanadium and niobium carbides, and finer grains. As a result of this work, the design stress of coil springs was raised from 900 MPa to 1000 MPa.

For the purpose of saving natural resources, and as a reaction to new automobile emission control regulations, requirements to reduce vehicular weight increased particularly after 1990. The design stress of coil springs was successfully raised to 1300 MPa using a 0.4% C-2.5% Si-0.8% Mn-2.0% Ni-0.85% Cr-0.4% Mo-0.2% V steel with a weight-saving of over 20% being achieved (17). However, owing to the high cost of the steel, which contains much more Ni, Mo and V than conventional spring steel, its usage has been decreasing.

In contrast, a new grade for 1200 MPa design stress has been developed and is gradually increasing in commercial production (18). The steel composition is typically 0.4% C-1.8% Si-0.5% Ni-1.1% Cr-0.15% V-0.025% Nb-0.0015% B. Carbon is reduced for increasing corrosion fatigue life, Si remains at a relatively high level for sag resistance, Ni is added to retard pitting corrosion, and Nb and B are added for grain refinement and strengthening of the prior-austenite grain boundaries.

Leaf Spring

Leaf springs are also expected to have high endurance with a thinner gauge. Ohmori (19) approached this subject using a modified ausforming process. In this process, hot rolling, in which leaf springs were reduced in thickness by 50-70% at 1123 - 1023° K, was followed by rapid quenching. Tapered leaf springs of specification JIS-SUP10, equivalent to 50CrV4, were produced by this process and revealed a better strength-toughness balance and higher fatigue strength to springs produced using conventionally quenched and tempered SUP10. The improved properties were attributed to refinement of martensite laths and improvement in carbide morphology in the matrix.

In a conventional layout of equipment used in mass production, however, it takes a comparatively long time to convey the rolled leafs to the quenching baths. Consequently, during the transferring period, the rolled leafs recover easily and internal strain, which affects the structural improvement, is reduced. Ohmori et al. (20) report that niobium additions to spring steel prolong the recovery time after rolling. Pawelski et al. also observed that the incubation time for austenite recrystallization after hot forming was retarded with 0.03-0.06%

Nb addition to 50CrV4 spring steel (21).

In a leaf springs production line, this beneficial effect of Nb has been demonstrated when the Nb and Ti added 50CrV4 steel was hot rolled, immediately quenched and tempered at 553°K (22). In comparison with the tensile strength of 1700 MPa in a conventionally hardened-tempered 50CrV4, the niobium bearing steel attained 2000 MPa in tensile strength and a 20% higher fatigue strength without any decrease in toughness.

Case Carburizing Steels

Case hardened automotive components, such as transmission gears and constant velocity joint-shafts, are produced through several processes and heat treatments and are used under highly loaded circumstances. For this reason the case hardening steels should satisfy three major properties. First; good formability, such as forgability and machinability. Second; (concerning the hardening process, such as ease of carburizing) desired hardenability and less distortion after quenching. Third; preferred mechanical properties after carburizing such as bending fatigue strength and impact endurance at the tooth root or around oil holes, and pitting resistance at the tooth face.

To fulfill these properties alloy design and processing sequence are optimized together with cost minimization. Figure 4 shows typical manufacturing processes for carburized gears. Case hardening steel bars are hot-forged, normalized, machined to a gear shape and then carburized and tempered. In addition, grinding and/or shot peening are conducted as the need arises. As a result, from the view of total cost reduction, cold forging has gradually taken the place of hot forging and has resulted in eliminating the costly hobbing process.

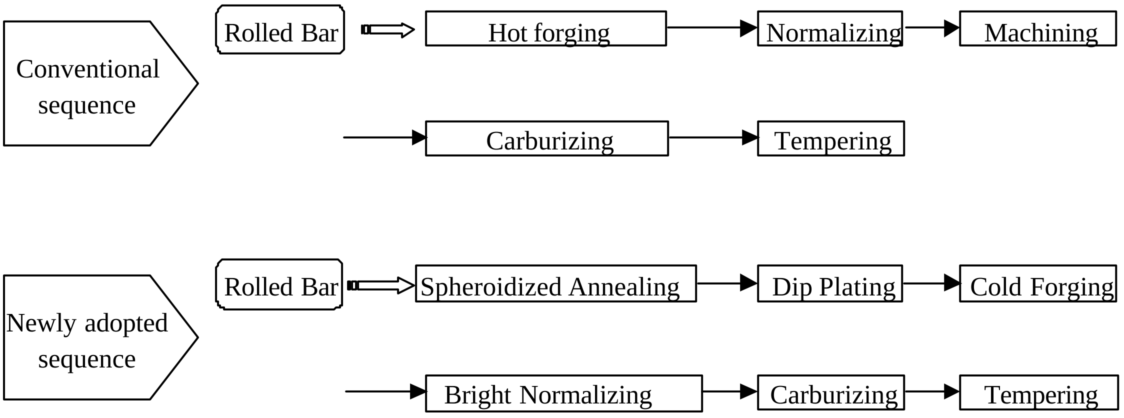

Figure 4: Typical manufacturing sequences for case hardened parts.

Measures Taken to Change Production Sequences for Carburized Gears

Usually the hardness of typical case hardening hot-rolled steel bars is too high for cold forging, thus, spheroidized annealing is necessary after rolling. Moreover, retained strain in cold-forged parts is a driving force leading to coarse grains and/or abnormal grain growth during carburizing at a high temperature. Bright normalizing in a vacuum in order to avoid a worsening of the surface quality of the cold-forged parts is also necessitated. Eventually the cost merit by introducing cold forging is offset by the cost of the newly added heat treatments.

Our target was to develop a case carburizing steel that had both sufficient cold forgability without the need for spheroidized annealing and which had resistance to grain coarsening during carburizing without bright normalizing. To achieve this goal, fundamental experiments were carried out to clarify the effect of alloying elements on the hardness in hot-rolled bars and the grain growth behavior in cold formed specimens (23, 24).

Prior to this project, we had already succeeded in developing cold formable carbon-boron steel with hot-forged CVJ parts made from JIS S48C and S53C, equivalent to SAE 1048 and 1053, having been substituted with the new grades (25). In this case, a small amount of B was added to increase hardenability in place of excess Si, Mn and Cr. This alloying design was applied to carburizing steel for cold forging.

Improvement in Cold Formability

The decrease in deformation resistance of parts cold formed is the most effective way to ensure the endurance of forming dies. Figure 5 presents the relationship between the hardness of specimens of JIS-SCM420 and SCr420, equivalent to 4118 and 5120, and deformation resistance at a true strain of 1.0 during cold forming at ambient temperature. Specimens were prepared from hot-rolled bars and from spheroidized annealed bars. It was clearly observed that the deformation resistance increases with the hardness of the specimens. In general, the hardness of steel with desirable cold formability, should be less than 80 HRB. However, it is hard to attain such a low hardness in as-rolled conditions, even after controlled rolling and cooling.

The effect of alloying elements on the hardness of hot rolled bars of base steels was examined. Chemical compositions of tested materials are shown in Table II. The base composition of the specimen was 0.20% C-0.07% Si-0.50% Mn-1.0% Cr. 32 mm diameter bars were normalized at 1200°K to simulate the hot rolling condition. Silicon and Mn and especially C, resulted in an increase in the steel hardness. In contrast, B did not have an obvious influence on the bar hardness. Here again, the combination of lowering C, Si and Mn contents together with B addition that compensates for the decline of hardenability was considered to be suitable for cold-forged steel.

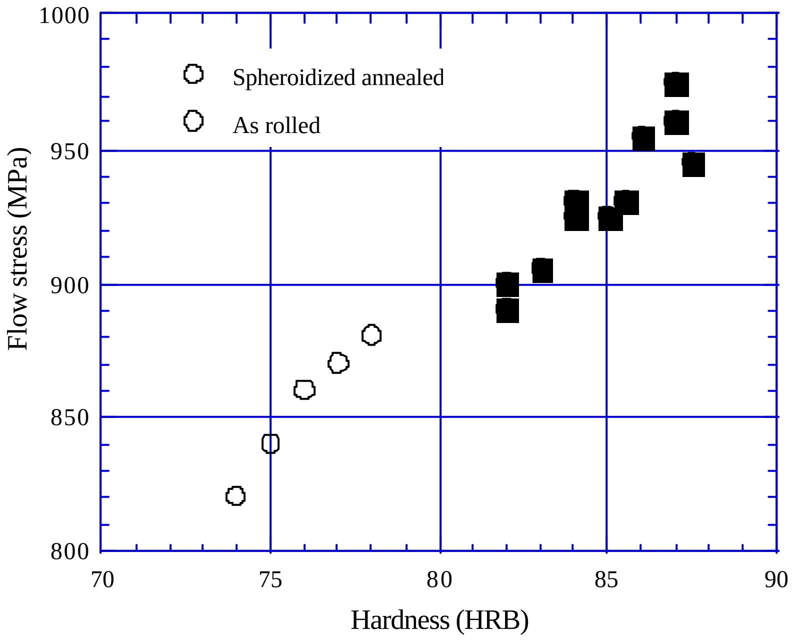

Figure 5: Relationship between flow stress during cold forming and hardness in steels.

Table II Range of chemical compositions of test steels (wt %)

Grade	C	Si	Mn	Cr	B	Others
Non-B	0.20	0.20	0.80	1.15	-	S:0.015, P:0.012
B	0.10-0.25	0.01-0.30	0.25-1.25	0.25-1.25	0.0015-0.0030	?

Prevention of Grain Coarsening During Carburizing

The most effective method to prevent grain growth in case hardening steels is to utilize precipitates of carbide, nitrides, and carbonitrides; in the case of Al-killed steels small aluminum nitride (AlN) particles suppress the migration of grain boundaries.

However, in B added case hardening steel, which would be expected to be applicable for cold forging, AlN precipitates cannot be used for pinning grain boundaries because B in solid solution combines with N in the steel prior to AlN precipitation. This BN precipitation leads to a lessening of the effect of B on hardenability. Therefore, Ti, which has a greater affinity to N than B, is added to fix the N as relatively coarse TiN precipitates. Finally, neither a small quantity of AlN, nor coarse TiN particles, can be considered as candidates to prevent grain growth in boron-bearing steel.

Fundamental studies on grain growth behavior in cold formed and reheated steels were examined. JIS-SCr420, boron steel, and 0.05% Nb added boron steel were 70% cold-rolled and heated at various temperatures for 1.8ks. Figures 6 and 7 respectively show average grain size number and area fraction of coarse grains, with the later defined as larger than ASTM grain size number 2. Figure 8 shows the optical microphotographs of each grade after reheating in the temperature range 1173 – 1273° K. The addition of B-only worsens grain growth behavior as previously described. On the contrary, the niobium- bearing, boron steel exhibits a finer grain than conventional case hardening steel.

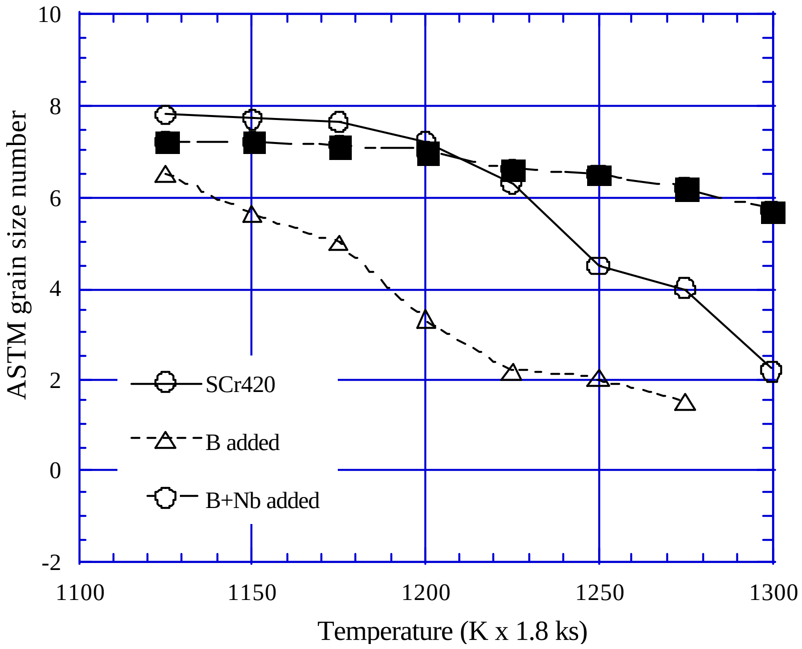

Figure 6: Grain coarsening behavior of specimens heated at various temperatures.

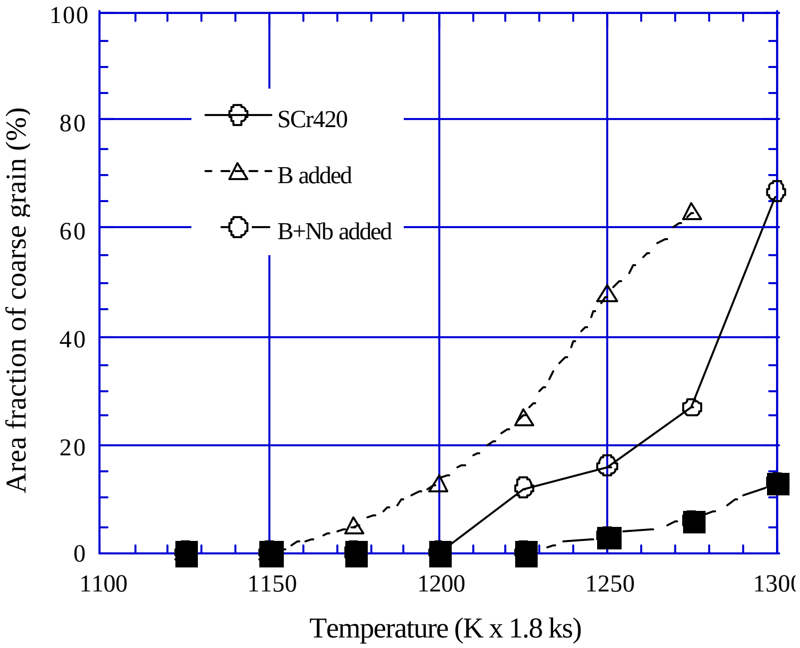

Figure 7: Area fraction of coarse grains in heated steels.

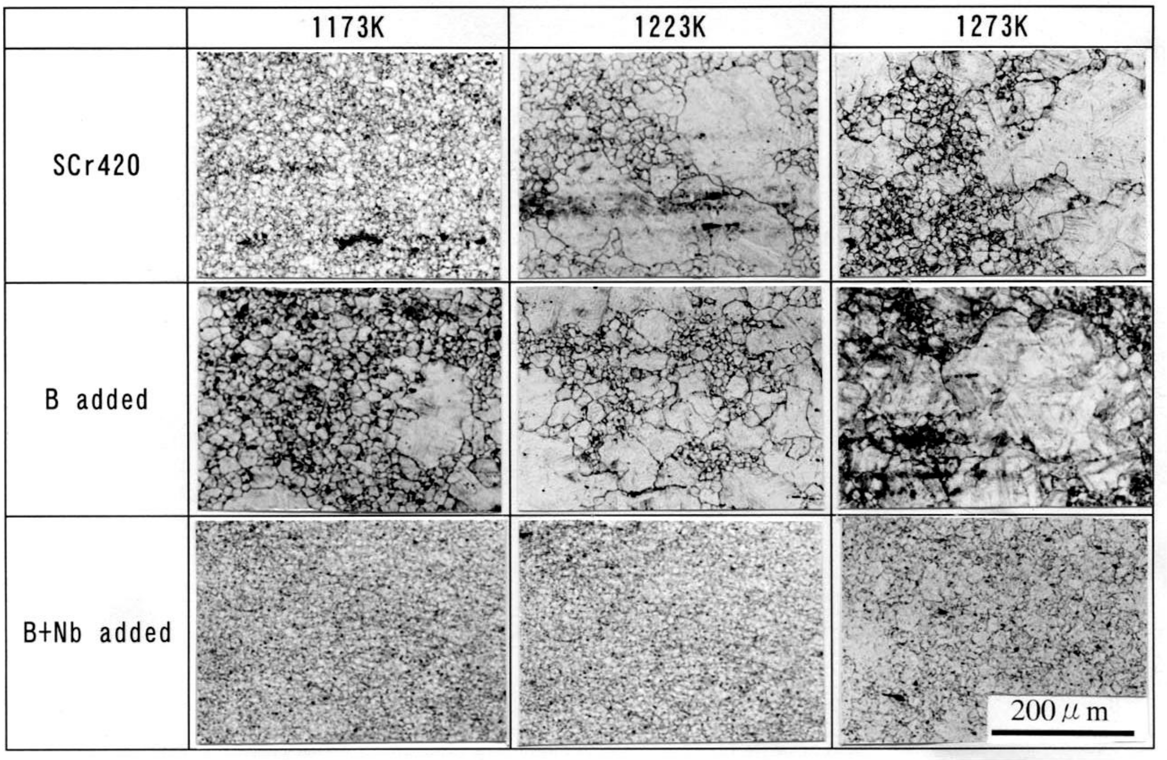

Figure 8: Optical micrographs of test specimens heated at various temperatures.

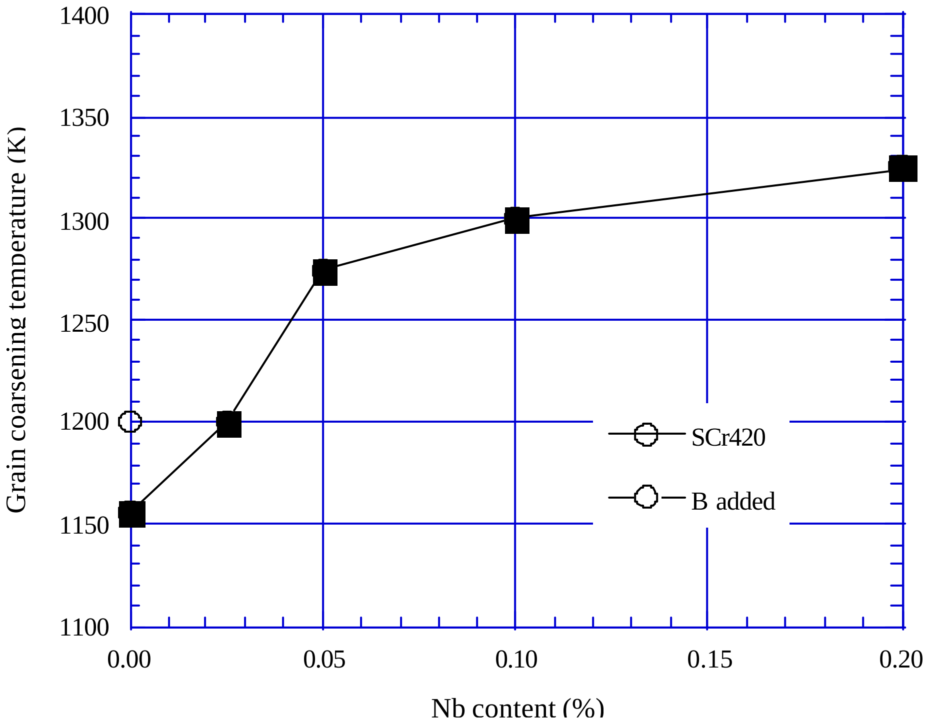

Figure 9: Effect of Nb content on resistance to grain coarsening.

Figure 9 illustrates the relationship between Nb content and grain-coarsening temperatures, where the area fraction of coarse grains is as high as 5%. It is clearly seen that the grain-coarsening temperature increases with Nb content and 0.05% of Nb is enough to retain fine grains even at 1273K. In practice, the amount of a niobium addition should be determined from the carburizing temperature and time at temperature according to each individual case.

Evaluation of the Developed Steel

The chemical composition of the developed steel was typically 0.2% C-0.10% Si-0.50% Mn-1.0 / 2.0% Cr-0.0015% B-0.05% Nb. In order to decrease the hardness of the steel after rolling, the C, Si and Mn levels were reduced. The hardenability of the steel was adjusted by the addition of boron and the level of chromium. Niobium was added to prevent grain coarsening during carburizing.

Two tons of billets were hot rolled and some of the rolled bars were spheroidized annealed. Figure 10 shows the hardness of the developed steels with 1.5% Cr content and conventional SCr420 steel. The hardness of the developed steel in the as-rolled state was 77 HRB and was almost equal to 75 HRB of the spheroidized SCr420. This indicates that the developed steel can be applied to cold forging without spheroidized annealing. Moreover, if the developed steel is spheroidized, the hardness becomes lower than 68 HRB which means that the new steel can be cold-forged to a more complicated shape. Compression tests were performed at room temperature to evaluate the cold formability of the steel. The hot-rolled developed steel showed a similar work hardening rate and superior critical strain for the prevention of crack initiation in the spheroidized SCr420.

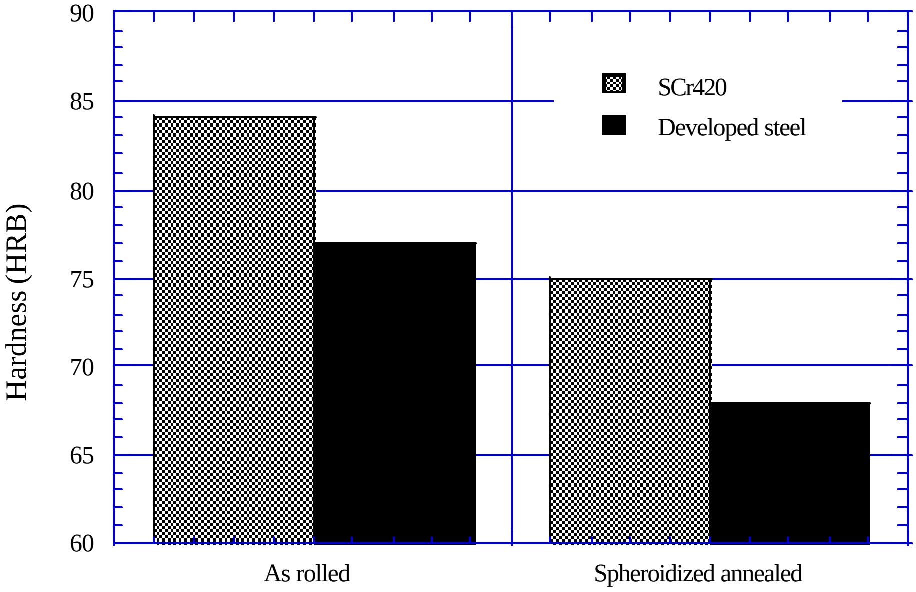

Figure 10: Hardness of developed steels and conventional case hardening steels (SCr420) in the as-rolled and spheroidized annealed conditions.

Figure 11 presents the grain coarsening behavior of the developed steel and SCr420 cold-forged steel up to 70% deformation and then heated at various temperatures for 1.8 ks. As expected, the developed steel exhibited finer grains at 1273°K than the SCr420 steel. Next, specimens of 25 mm diameter and 130 mm length were carburized at 1183°K and quenched into a 413°K oil bath to investigate the carburizing properties of the steels. The developed steel had a surface hardness of 750 HV and its microstructure consisted of a tempered martensite phase with no troostite.

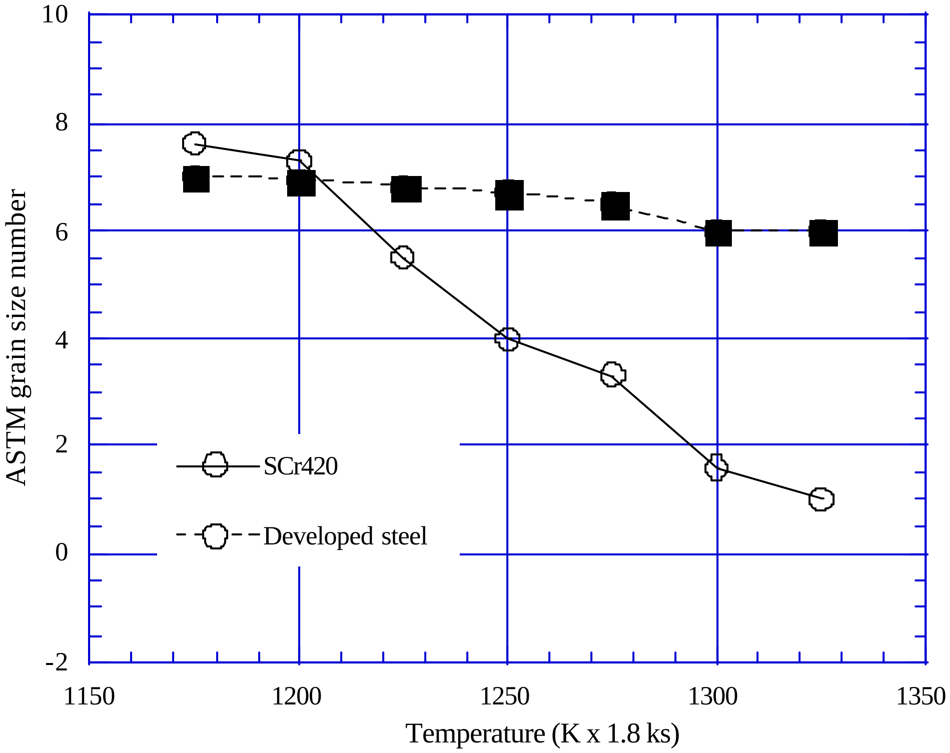

Figure 11: Grain coarsening behavior of the Nb-containing developed steel and SCr420 cold-forged up to 70% and then heated at various temperatures.

Fatigue properties of carburized specimens are listed in Table III. The rotating bending fatigue specimens with a notch of 1.84 stress intensity factor were carburized at 1183°K for 3 hours, oil-quenched from 1103°K and tempered at 433°K. Since shot peening is becoming popular to improve the fatigue strength in carburized parts, some of the specimens were shot peened at 0.7 mm arc height intensity. The Nb-contained developed steel has a fatigue strength equivalent to the conventional SCr420 steel at any surface condition.

Table III: Comparison of mechanical properties of the developed Nb-containing steel and SCr420

	Rotating bending fatigue strength		Cycles to pitting
	As carburized	Shot peened	As carburized
SCr420	500 MPa	820 MPa	2.3-9.0 x 107
Developed Steel	510 MPa	810 MPa	1.5 - 8.3 x 108

Pitting resistance was evaluated using geared roller testing machines. In this test a rotating 26 mm diameter specimen was contact loaded by a 130 mm diameter disk. The contact stress was 2.94 GPa and the slip ratio was 40 percent. Automatic transmission fluid was used as the lubricating oil at 353°K. The Nb-containing steel attains the same pitting resistance as the conventional steel.

Charpy-impact specimens with 10-mm R notch were carburized and tested to compare toughness. As shown in Figure 12, the impact value of the developed steel was superior to a NiCrMo steel, such as JIS-SNCM420 (SAE4320).

At the present time, the developed Nb-added boron steel and a niobium-bearing SCM420 grade (26), the latter has higher hardenability than the former, are being manufactured at the rate of 500 tons per month for cold-forged automotive components.

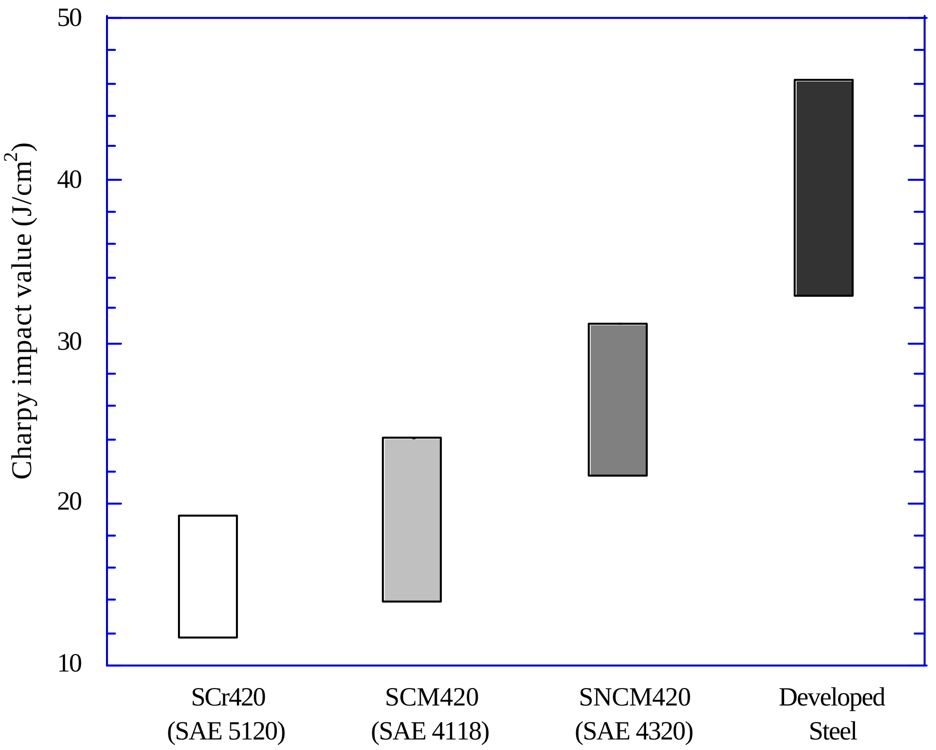

Figure 12: Impact properties of the Nb-containing developed steel and the conventional case hardening steels after carburizing.

Summary

Thus far, structural refinement and precipitation hardening by the addition of Nb has been made use of in the development of "non heat-treated" steels for forged parts and fasteners, which were previously produced from quenched and tempered grades. At the same time the strong effect of niobium precipitates on restricting grain coarsening in "heat-treated" steels has become popular. The new niobium bearing heat-treated grades for high strength fasteners, springs and case hardened parts contribute to the strengthening of automotive components and/or contributing to a saving in production costs and energy.

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