EFFECTS OF Nb, V AND Ti ON THE EVOLUTION OF STRUCTURE IN MEDIUM CARBON STEELS DURING VARIOUS HOT FORGING STEPS

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Abstract

Microalloying of medium carbon steels by Nb, V and Ti is applied for obtaining desired properties without additional heat treatment of forgings. Hot forging of long products is confined by heating the blank and hot deformation followed by air or controlled cooling. Additions of microalloying elements affect the rate of grain growth during heating blanks, recrystallization parameters of deformed austenite, temperature of gamma-alpha transformation at cooling as well as contribute to precipitation hardening of the ferrite constituent. There are numerous publications on this subject; however there are not many systematical investigations that compare the effects of Nb, V and Ti quantitatively. The current presentation is aimed at summarizing the effects of Nb, V and Ti on processes of structure evolution, parameters of final ferrite-pearlite structure as well as on the mechanical properties of medium carbon steel containing manganese and chromium.

Introduction

Forged components made from medium-carbon steels microalloyed by vanadium, have come into use in the automotive industry since the beginning of the 1970s aiming to replace quench and temper (Q&T) to reduce cost as well as time and energy consumption [1-3]. Typical compositions of the forging steels contain about 0.3-0.6% carbon necessary for strength of 800-1000 MPa, comparable with heat treated steels. The steels also contain manganese (and chromium) for both strength and structure control. Aiming for better machinability than that of corresponding heat treated steels, it is desirable to have the final structure consisting of ferrite plus pearlite, so microalloying by V, Nb or Ti has a critical role to provide substantial contribution to the strength of steels due to grain refinement and precipitation hardening. Structure optimization is very important to keep the necessary balance of strength, impact toughness and brittle to ductile transition temperature to enable these steels to replace heat treated ones.

There are numerous publications and patents offering quite reasonable solutions and experimental data explaining effects of microalloying elements in steels for forging without heat treatment [4-6], however there is insufficient literature to compare different microalloying elements and demonstrate their combined effects. Therefore, the current paper
is aiming to summarize experimental data on the matter obtained by the author jointly and a few Ph.D. students [7,8].

**Materials and experimental procedure**

Composition of investigated steel contained mostly 0.3C-1Mn-0.6Cr-0.5Si (designated further as 30C1Mn1Cr) with widely variable content of Nb, V and Ti that were added individually and in various combinations. Laboratory heats were melted in 60- and 150 kg air induction furnace. Ingots of lab heats were deformed to 50x60 mm billets that were used as objects for investigations.

The cycle simulating the forging process starting from those billets is presented in Figure 1a. It is clear that the final microstructure and corresponding properties of forgings will be controlled by four steps of structure evolution: (a) grain growth during preheating of billets; (b) recrystallization kinetics of deformed structure; (c) transformation of austenite during cooling; (d) precipitation hardening that can take place during deformation and final cooling.

Grain growth of compared steels at preheating up to 1200°C was investigated using vacuum etching technique at soaking times for 30 minutes at compared temperatures. Recrystallization of deformed austenite was studied by the tensile testing method based on measured softening ratio R [9] after isothermal holding following to deformation with 20-30% reduction (see Figure1b).

![Figure 1. Experimental cycle used for simulation of forging process (a) and diagram of preheating/loading/isothermal holding/unloading to measure softening ratio R (b).](image)

\[ R = \frac{\sigma_{20} - \sigma''}{\sigma_{20} - \sigma'} \times 100\% \]

where \(\sigma_{20}\) – flow stress at 20% deformation, \(\sigma'\) - yield stress before isothermal holding, \(\sigma''\) - yield stress after isothermal holding.
Precipitation hardening as well as changes in pearlite morphology was investigated thoroughly by TEM using both replicas and foils.

**Effect of microalloying elements during pre-heating and deformation**

The properties of microalloyed steels are basically determined by the volume and size of microstructural constituents and precipitation hardening. The initial preheating temperature dictates the properties through combined effects of the initial (prior to deformation) grain size and sizes of structure elements after deformation and finally, after phase transformation. Therefore the systematic study of the influence of microalloying elements on susceptibility of steels to grain growth at various temperatures of austenitization is very important. Figure 2 illustrates the effects of compared microalloying elements on grain sizes at different temperatures of austenitization.

![Figure 2. Effect of microalloying elements in 0.3C-1Mn-1Cr steel on austenite grain size at various temperatures of austenitization.](image)

As expected, V retards the growth of austenite grains during heating to a lesser extent than Nb and Ti because of its high solubility. Nb appeared to be the most effective in refining structure albeit it results in some grain heterogeneity at 1200°C preheating temperature.

Figure 3 shows changes in the softening ratio, R, in dependence on the content of microalloying elements, austenitizing temperatures, temperatures of deformation and isothermal holding time after deformation. As shown for both austenitizing temperatures, 1150°C and 1200°C, titanium demonstrates the least retardation of recrystallization whereas niobium is the highest regardless of post-deformation isothermal holding time.
Figure 3. Effect of microalloying elements on softening ratio $R$ at various temperatures of deformation of 30C1Mn1Cr steels.
- .03%Ti; △ -0.05%V; ▲ - 0.1%V; ○ - 0.045%Nb; ● - 0.14%Nb

This is due to the fact that the temperature range of deformation coincides with the temperature range of niobium carbonitride precipitation (900-1000°C). For vanadium this temperature range of precipitation is lower, so that efficient retardation of recrystallization by vanadium takes place at temperature of deformation below 900°C.

The effect of initial austenitizing temperature on the retardation of recrystallization in the hot-deformed austenite depends on the solubility of the specific microalloying elements and the following opportunity of its precipitations during deformation. Thus, an elevation of the austenitizing temperature in niobium-bearing steels leads to an enhancement in retardation of recrystallization. For example, at $T_a=1150^{\circ}C$ suppression of recrystallization for steel containing 0.14%Nb is possible only at $T_{def}$ below 900°C, however at $T_a=1250^{\circ}C$ it takes place at $T_{def}=950^{\circ}C$ and at substantially lower Nb content = 0.045%. For vanadium steels an elevation of the austenitizing temperature leads to significant retardation of recrystallization only for deformation temperatures below 900°C, when precipitation of vanadium carbonitride can be observed. For titanium-bearing steels, the effect of elevated austenitizing temperatures appeared to be substantial only in the case of short post-deformation holding times (10 sec) and at deformation temperatures below 900°C. At holding time of 1 minute in steel with 0.03%Ti grain growth takes place that results in $R>1$. 

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Grain growth and increase in soluble content of microalloying elements can also result in increase in incubation time of recrystallization after deformation. Since processes of precipitation and recrystallization compete and beginning of one prevents other [10], increase in incubation time of recrystallization creates the opportunity for carbonitrides to precipitate on grain boundaries of deformed grains. Similar effect was observed for Nb that, in turn, additionally enhances retardation of recrystallization.

The higher effect of retardation of recrystallization due to higher content of V or Nb is bound to higher volume of precipitates induced by deformation. However, this is valid only within the limits of solubility. As shown in Figure 4, at Nb content above the limit of its solubility the coalescence of particles and decrease in their number at 1150°C can result in a lower retardation effect.

![Figure 4. Effect of Nb content on softening ratio, S, of 30C1Mn1Cr steel deformed at 1000°C.](image)

**Effect of microalloying elements on parameters and products of austenite transformation**

The next important factor that determines the properties of microalloyed steels after forging is the type and morphology of microstructure formed after transformation of deformed austenite. Investigation of effects of Nb, V and Ti on phase transformation and final structure was performed using high speed deformation dilatometer Barr 805.

As found, microalloying additions do not affect parameters of $\alpha$-$\gamma$ transformation at heating: $A_{c1}$ temperature varied from 740 to 760°C, while $A_{c3}$ ranges within 820 and 840°C. Quite different effects were observed at cooling with cooling rate of 1°C that is corresponding to the average still air cooling rate of a forging of several kilograms mass at heating: $A_{c1}$ temperature varied
from 740 to 760°C, while \( A_{c3} \) ranges within 820 and 840°C. Quite different effects were observed at cooling with a cooling rate of 1°C that is corresponding to the average still air cooling rate of forging of several kilograms mass. As shown in Figure 5, all microalloying elements contract the temperature range of ferrite formation that proceeds with very high intensity though. Since no precipitate was observed during deformation, the visible increase in stability of austenite by Nb and V (decrease in \( T_{fs} \)) can be explained by their presence in solid solution. Up to the content of ~0.05%, close to its limit of solubility at 1150°C, Nb is decreasing \( T_{fs} \); at further increase in Nb content the existing niobium carbonitrides evidently facilitate ferrite nucleation (Figure 6).

Figure 5. Effect of microalloying additions on temperatures of ferrite start (\( T_{fs} \)), pearlite start (\( T_{ps} \)) and finish (\( T_{pf} \)) at continuous cooling of 30C1Mn1Cr steel.
Figure 6. A string of Nb carbonitrides precipitated on boundaries of prior austenite grains serve as centers of ferrite nucleation.

Ti is practically insoluble at 1150°C austenitizing temperature; therefore it does affect the temperature of ferrite formation. The process of \( \gamma \rightarrow \alpha \) transformation initiates the precipitation of vanadium and niobium carbonitrides, leading to a decrease in the stability of the residual austenite portion and a relative increase in \( F_{ps} \) temperature. In titanium-bearing steels it is likely that the observed increase in pearlite start temperature is only associated with austenite grain refinement. Effect of microalloying elements on the pearlite formation temperature range is varying: vanadium contracts it, while niobium and titanium broaden it. What is important, niobium contributes to lowering the pearlite transformation finish temperature by 30–40°C.

Deformation of austenite substantially affects the kinetics of austenite decomposition. In the present investigation, this effect was studied using dilatometry on 30CMn1Cr1VNb steel after austenitization at 1150°C, compression at 850°C on 30% with a strain rate of 0.8sec\(^{-1}\). It was found that deformation promotes diffusion products of austenite decomposition (Figure 7). Thus, cooling at 1°C/sec after deformation results exclusively in ferrite-pearlite mixture, while cooling of undeformed austenite at the same rate produces almost completely bainitic structure. Increase in \( A_{1} \) and \( A_{3} \) was also observed. All those changes in transformation of deformed austenite in investigated microalloyed steels are associated with an increase in structural imperfections resulting from work hardening of the austenite (when recrystallization is suppressed) and from the precipitation of the carbonitride phases in the temperature range between the end of deformation and before transformation.
Effect of V, Nb, Ti on final structure and precipitation hardening

The comparative investigation of effects of vanadium, niobium and titanium content on final structure and properties was performed using steel with a base composition of 0.3% carbon, 1.0% manganese, 0.5% silicon, 0.6% chromium and 0.018% nitrogen*. The steels were austenitized at 1200°C, subjected to 70% deformation at 900°C and cooled at a rate of 1°C/sec. Considering the fact that the volume fraction of pearlite in medium-carbon steels is as a rule greater than 50%, it is evident that factors relating to quantity of pearlite and its morphology appeared to be of the most important. Therefore, the volume fraction of pearlite, $V_p$, and pearlite colony size, $D_p$ (length of the average chord), were measured as critical microstructural parameters as well as interlamellar spacing and cementite lamella thickness. The dependence of $V_p$ and $D_p$ on vanadium, niobium and titanium content is illustrated in Figure 8.

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1 Results presented in [8] have shown advantages of the increased content of nitrogen that result in preferred carbonitrides formation vs. carbides. Carbonitrides are featured by less solubility and wider temperature range of precipitation in favor of larger volume of finer particles that, in turn, promote structure refinement.
An increase in content of V and Ti as well as Nb after ~0.05% leads to a decrease in the volume fraction of pearlite. The value of the decreased pearlite volume fraction exceeds the value calculated on the basis of carbon tied in carbonitrides or even carbides and is associated with the decreased stability of the austenite due to the growing number of structural defects prior to the $\gamma$-$\alpha$ transformation. The apparent reduction in pearlite volume fraction in the titanium-bearing steel is observed only at Ti content that assumes that all of the nitrogen is tied up in nitrides, however, here as well the decrease in pearlite fraction $V_p$ is greater than can be explained by reduced carbon content due to carbide formation. The pearlite colony size $D_p$ depends to a great extent on the titanium and niobium contents, particularly in the case of niobium at levels that exceeded the solubility limit (0.05%), as a result of strong retardation of recrystallization.

The performed measurements of ferrite microhardness had shown that the greatest precipitation hardening is brought by carbonitrides of vanadium, followed by niobium and lastly titanium. Pearlite microhardness exhibits a more complex dependency on microalloying element content. For example, an increase in the vanadium content leads to an increase in pearlite microhardness both because of refinement of the pearlite and precipitation hardening of ferrite component of pearlite. The maximum pearlite microhardness in the niobium-bearing steel occurred at 0.05% niobium, which corresponds to the point of maximum austenite stability and lowest pearlite formation temperature. As a whole, pearlite in the niobium steel exhibits lower microhardness than that in the vanadium steel because of less precipitation hardening of the ferrite lamellae and because of the relatively higher temperature of pearlite starting transformation.

The effect of titanium is more complex because titanium forms carbides, carbonitrides and nitrides, all differing in solubility and size. Nevertheless the whole effect of titanium can be explained on the basis of effects which are similar to vanadium and niobium.

Three types of carbonitrides can be distinguished in all investigated steels: large particles not dissolved in the austenite during soaking prior to deformation; particles precipitated during deformation, and particles precipitated during subsequent decomposition of the austenite. Titanium nitrides and carbonitrides of niobium are examples of the first type; their sizes range from 0.1-1.0 $\mu$m, they have cubic shapes and are randomly distributed.
Vanadium and niobium carbonitrides precipitate during deformation or immediately thereafter. Vanadium carbonitrides on subgrain boundaries have the appearance of chains composed of particles with sizes up to 600A, while niobium carbonitrides appear in multi-row chains and range in size from 100 to 2000A. Coarsest niobium carbonitride particles, as a rule, are located on former austenitic grain boundaries and become more refined at increase in their distance from the boundaries. This difference in sizes can probably be explained by different precipitation temperatures and by the difference in diffusion rates along the grain boundaries and within the grains themselves.

The sizes of vanadium carbonitrides are practically the same in austenite as in ferrite (100-500A), however those precipitated in austenite are roughly cubic and are less transparent to electrons than the disk-like precipitation in ferrite. By contrary, niobium carbonitride precipitates are considerably finer in ferrite than in austenite.

In addition to the discrete carbonitride particles formed in proeutectoid ferrite, some niobium carbonitrides were also found in clusters and as individual particles in ferrite lamellas in the pearlite. The whole range of sizes of particles in the niobium-bearing steel varied from 20 to 3000A; in vanadium-bearing steel from 10 to 600A, and those in the titanium-bearing steel, from 500 to 5000A.

Important to note that particles in vanadium- and titanium-bearing steels were demonstrated a relatively continuous range of sizes, whereas the precipitates in the niobium steel appeared to be grouped in two size ranges: smaller than 100 A and larger than 1000 A. Intermediate sizes were not observed in this steel. Microstructural analysis of the steels microalloyed by combination of investigated elements had revealed the formation of vanadium-niobium and niobium-titanium carbonitrides. Vanadium-niobium carbonitrides are slightly coarser than the “pure” vanadium carbonitride and are precipitating over a wider range of temperatures and therefore, better able to retard recrystallization and facilitates decomposition of austenite. As a result more significant refinement of the final structure and more uniformity can be observed in steels with combinations of Nb and V that promotes both higher strength and toughness.

In a combined addition of titanium and vanadium, the titanium's strong affinity to nitrogen reduces the volume fraction of vanadium carbonitrides. While it is theoretically possible to get only vanadium carbides after totally binding the nitrogen by titanium, in practice, vanadium carbides do not have sufficient time for precipitation during air cooling. As a result, adding titanium in V-Ti steels results in decrease in strength and a corresponding increase in toughness. Relationships between strength and toughness of compared steels with various combinations of additions of microalloying elements are illustrated in Figure 9.
Figure 9. Relationship of tensile strength, impact toughness and FATT for steels containing various combinations of microalloying elements to 30C1Mn1Cr base composition.

- V-Nb combination
- V-Ti combination
- Nb-Ti combination

In Figure 10 we can see relationship of steel composition, structure (volume fraction of pearlite, $V_p$) and properties of investigated steels. Arrows indicate direction of increase in microalloying content, numbers show sizes of pearlite grains, $D_p$. Volume of fraction of pearlite in investigated steels is ranging from 48 to 64% indicating that it is diluted by carbon that should result in its relatively lower strength and higher toughness.
As known, the strength of pearlite strongly depends on its interlamellar space $S_0$, but its toughness – mostly on the thickness of cementite plates, $t$. Quantitative investigation of these parameters was performed using carbon replicas\(^1\). Figure 11 on the next page demonstrates data that allow for distinguishing the changes in $S_0$ and $t$ in dependence on the content of microalloying elements.

\(^1\)This method gave increased values of plate thickness but allowed for seeing trends of changes.
Figure 11. Dependence of thickness of cementite thickness, $t$, and interlamellar spacing $S_o$ on content of microalloying elements in 30CMn1Cr steel.

As known $S_o$ strongly depends on the temperature of formation of pearlite being an inverse proportion to $\Delta T$ under-cooling below the eutectoid temperature [11]. Therefore all microalloying elements mostly decrease $S_o$, but Nb only before 0.045%. In contrast, Nb is the only element that decreases the thickness of cementite plate thickness promoting higher toughness of steel. Based on that data it is easier to understand advantages of combined additions demonstrated earlier in Figure 9.

Conclusions

It follows from the above observations that the best combination of properties results first from an optimum austenitizing temperature, necessary for the maximum dissolution of microalloying elements, but without undesirable grain growth, followed by deformation at a temperature that is sufficiently low in order to prevent recrystallization of austenite before transformation.

Based on comparison of effects of Nb, V, and Ti on grain growth at pre-heating, retardation of recrystallization, sizes of precipitation as well as on sizes of grains and fine structure of pearlite the best balance of strength and toughness of medium carbon as-forged steels can be achieved using combination of Nb and V.

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References


