EFFECT OF INOCULATION ON MICROSTRUCTURE AND PROPERTIES OF LOW C-Mn AND LOW ALLOY STEELS

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Abstract Effect of the addition of various inoculants on the morphology and mechanical properties of low C-Mn and low alloy steel samples is studied by thermal and mechanical processing of the samples after solidification. According to the results obtained from metallographic studies with the electron microscope and microanalysis with x-rays, the distribution of inoculating agents in the steel matrix is seen in the form of very fine precipitates dispersed in the austenite-ferrite microstructure. These precipitates retard low-temperature recrystallization of austenite, increase the specific boundary area of retained grains and enhance concentration of more favored nucleation sites when austenite to ferrite transformation occurs at the final cooling stage from normalization temperature and thus cause a recognizable refinement in the steel microstructure. Both the strength and hardness of the samples are considerably enhanced by increasing the content of the inoculating agents, while the decrease in the elongation of the samples is limited to an acceptable range.

چکیده تاثیر افزودن عوامل تلقیح شونده گوناگون بر ساختار و خواص مکانیکی فولادهای کم کربن ـ منگنزی و کم آلیاژی از طریق عملیات حرارتی و مکانیکی بعد از انجماد، تحقیق شده است. براساس نتایج بدست آمده از طریق متالوگرافی با میکروسکوپ الکترونی و میکروآنالیز با اشعه ایکس، دیده می شود که توزیع عوامل تلقیح شونده در فولاد به صورت پخش رسوبات بسیار کوچک در ریزساختاری از آستنیت و فریت اتفاق می افتد. این رسوبات باعث کند شدن تبلور مجدد آستنیت در دماهای پائین می شوند، سطح ویژه مرز دانه های باقیمانده را افزایش می دهند و غلظت محلهای مناسب برای جوانه زنی در هنگام تغییر حالت آستنیت به فریت در مراحل سرد کردن نهایی از دمای نرمالیزاسیون را زیاد می کنند و نتیجتاً باعث ظریف شدن قابل ملاحظه ریز ساختار فولاد می شوند. بدین ترتیب هم استحکام و هم سختی نمونه ها با افزایش محتوای عوامل تلقیح شونده زیاد می شود و این در حالی است که کم شدن ازدیاد طول نسبی نمونه ها در محدوده قابل قبولی باقی می ماند.

INTRODUCTION

The development of high strength, low-alloy steel in the last 20 years has raised many new technological questions concerning its production, processing and application. The addition of a number of so called microalloying elements as inoculating agents to the steel with the purpose of 1) minimization of anisotropy in mechanical properties through formation of non-deformable inclusions, 2) refinement of structural austenite, ferrite and pearlite grains, 3) optimization of austenite/ ferrite interface morphology, 4) lowering of ductile to brittle transition temperature and 5) strengthening due to micropericipitation and/ or solute drag effect, has been the subject of much discussion [1-4].

There is, however, a lack of information about some areas of practical significance. Diffusion controlled carbonitride precipitation at lower austenite/ upper ferrite temperatures, thermodynamics and kinetics of austenite to ferrite transformation during simultaneous cooling and rolling operations, microalloying influence in nonisothermal deformation as compared with the isothermal process and inclusion shape control during the forging operation are a few examples which will be examined here.

In this study the effect of dispersion of small microalloying precipitates before, during and after austenite to ferrite transformation on mechanical properties of as-cast (AC), cast and normalized (CN), cast quenched and tempered (CQT), as-rolled (AR), rolled quenched and tempered (RQT), and forged quenched and tempered (FQT) steels is investigated. Two types of low-carbon manganese steels and low alloy steels are chosen for better inclusion control and formation of polygonal ferrite and reduced pearlite microstructures. Substoichiometric amounts of vanadium, niobium and rare earth elements are used as some typical microalloying agents with different solute drag, precipitation and chemical effects. The influence of the addition of these elements on mechanical properties of steels is discussed through microstructural evaluation of the samples.

METHODS AND RESULTS

The samples are prepared by melting steel in an alumina crucible and then casting it in a pre-heated iron mold. The preheating is to avoid surface hardening of the as-cast samples. The so called plunging technique is used to inject predetermined amounts of ferro-vanadium, ferro-niobium and mischmetal into the Al-killed liquid steel. The experimental details are described elsewhere [5]. The chemical compositions of the samples are given in Table 1.

The effect of V and Nb on the prior-

Table 1. Chemical Composition of Samples

Sample	Mean Chemical Composition (Wt %)														
	C	Si	Mn	Cr	Ni	Mo	Ce	La	Nd	V	Nb	Al	S	P	N*
1	0.42	0.50	1.03	1.27	-	0.213	-	-	-	-	-	0.13	0.022	0.040	
2	0.40	0.46	0.92	1.29	-	0.204	0.032	0.016	0.011	-	-	0.12	0.018	0.036	
3	0.30	0.41	0.84	0.99	-	0.176	0.042	0.022	0.015	-	-	0.14	0.017	0.035	
4	0.26	0.50	0.86	0.97	-	0.164	0.049	0.025	0.018	-	-	0.14	0.020	0.033	
5	0.48	0.57	0.84	1.20	1.75	0.310	-	-	-	0.12	-	0.10	0.020	0.018	
6	0.48	0.57	0.84	1.20	1.75	0.310	0.048	0.024	0.017	0.12	-	0.10	0.020	0.018	
7	0.48	0.57	0.84	1.20	1.75	0.310	0.060	0.030	0.021	0.12	-	0.10	0.020	0.018	
8	0.48	0.57	0.84	1.20	1.75	0.310	0.072	0.037	0.026	0.12	-	0.10	0.020	0.018	
9	0.042	-	0.26	0.12	0.15	-	-	-	-	-	-	-	0.031	0.016	0.006
10	0.039	-	0.25	0.12	0.11	-	-	-	-	0.06	-	-	0.024	0.015	0.006
11	0.034	-	0.21	0.11	0.11	-	-	-	-	0.12	0.025	-	0.020	0.015	0.006
12	0.120	-	0.72	0.04	0.02	-	-	-	-	-	-	-	0.023	0.010	0.008
13	0.140	-	0.85	0.04	0.03	-	-	-	-	0.14	-	-	0.023	0.010	0.008
14	0.130	-	0.72	0.04	0.03	-	-	-	-	0.18	0.14	-	0.020	0.011	0.008
15	0.140	0.02	0.89	0.05	0.03	-	-	-	-	0.22	0.22	-	0.019	0.011	0.008

** Estimated.*

austenite grain size determined by optical micrography of the AC, CN, CQT, AR, and RQT samples is demonstrated in Figure 1. Normalization is accomplished by retaining the samples at 910°C for 15 minutes and then cooling them in ambient temperature. The quenched-tempered specimens are prepared by retaining the steel in 910°C for fifteen minutes, quenching in water, reheating to 600°C, retaining for 15 minutes and quenching in water again.

Rolling starts at 910°C which is a little above the austenite to ferrite transformation temperature and finishes at 650°C which is below transformation temperature. This procedure is done in 10 stages with a residual strain of around 15 percent per stage. Forging is done by hammering samples 1 to 8, 30 times at 1200°C to 1/4 of their original cross sectional area. Longitudinal and transverse specimens are then prepared for tensile and impact tests. The specimens are heated in molten salt at 850°C for 10 minutes, quenched in oil and tempered at 350°C for 1 hour.

ASTM standard tensile test with a strain rate of 0.0017 sec^{-1}, Vickers pyramid hardness test and Sharpy impact test are utilized to determine the mechanical properties of the samples. The results are illustrated in Figures 2, 3 and 4 and Tables 2 and 3.

Replica electron microscopy is conducted on CQT, RQT and FQT specimens in order to assess the effect of reheating and deformation on the state of precipitation. The precipitation observations show a relatively uniform distribution of fine precipitates ranging in size from about 0.05 to 5 microns in both CQT and RQT specimens. The average size of the precipitates increases, however, with the nonisothermal deformation of the samples (Figure 5 (a) and (b)). Optical micrographs of FQT specimens in longitudinal and transverse directions show the lengthening effect of hot forging on MnS inclusions (Figure 6 (a) and (c)). Replica electron microscopy and x-ray microanalysis indicate an almost complete conversion of soft MnS inclusions in sample to nondeformable Rare Earth Metal (REM) oxy-sulfides in sample 4.

DISCUSSION

Despite the absence of strain hardening and application of low-temperature thermal treatment, complete recystallization in prior-austenite grains is observed in the non-deformed samples without microalloying elements. The retardation of grain growth due to the pinning effect of fine dispersions during the normalization process at temperatures well below the dissolution point of precipitates [6 and 7] results in reduction of grain size in the inoculated V and V+Nb steel samples (Figure 1). The data show that with the amount of N present in the samples, the effect of both V and

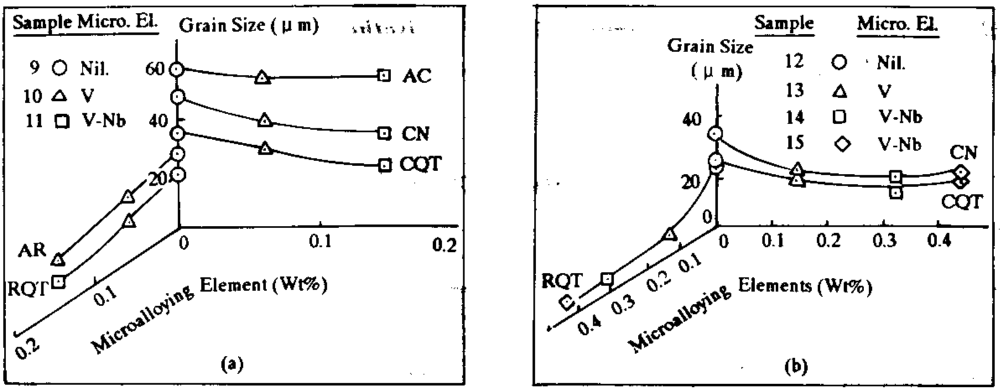


Figure 1. Austenite grain size after casting, rolling and heat treatment of steel (a) samples 9 to 11 and (b) samples 12 to 15.

Nb in reduction of the prior-austenite grain size is almost to the same extent. Nb combined with V shows, however, a reverse effect in the case of AC specimens (Figure 1 (a)) and CN and CQT samples with microalloying contents of greater than 0.3 percent (Figure 1 (b)). These effects can be easily attributed to the changes in the volume fraction and particle size of the carbonitride precipitates at the austenitising temperatures.

The data given in Figure 2 indicate that highly enhanced strength can be obtained with almost negligible toughness deterioration when nonisothermal rolling at extremely low temperatures, starting in the austenite and finishing in the ferrite region (910 - 650°C), is excersized with V and V + Nb steel samples. Production of fine ferrite grains, as a result of the deformation below recrystallization temperature can be of some interest. This seems to be an economically feasible method for

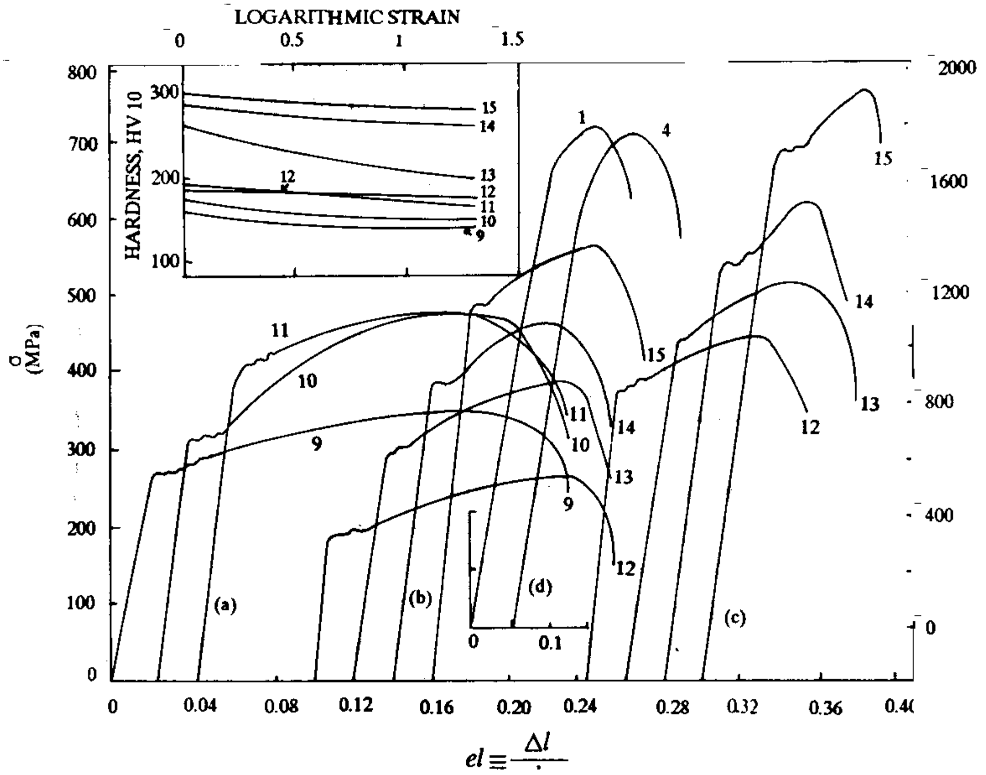


Figure 2. Stress-strain curves for (a) RQT samples 9 to 11, (b) RQT samples 12 to 15, (c) AR samples 12 to 15 and (d) FQT samples 1 and 4. Curves 1 and 4 read with right vertical axis. The origin of the horizontal axes is shifted for different curves. Effect of nonisothermal deformation on the hardness of quenched- tempered steel samples is also shown in the upper-left part of the diagram.

Table 2. Effect of Inoculation on Hardness and Sharpy Shelf Energy of Steel Samples.

Process	Hardness, HV 10							Process	Sharpy Shelf Energy, J	
	9	10	11	12	13	14	15		1	4
AC	121	148	137	168	252	283	279	FQT	Longitudinal	Longitudinal
CN	106	112	121	155	178	193	215		8	12
CQT	159	169	184	191	263	290	304		Transverse	Transverse
RQT	137	148	171	170	201	264	280		6	7.5

production of steel plates suitable for ship building and line pipe manufacturing purposes [8].

Prior hot rolling of austenite can cause the formation of ferrite nucleation sites and enhance the rate of transformation of austenite. Recovery and recystallization of deformed ferrite may thus result in good toughness accompanied by a high yield and tensile strength (Table 3). The nonisothermal deformation can, however, reduce this effect and cause the formation of a greater amount of pearlite (relative to ferrite and martensite) in the case of AR and RQT specimens as compared to AC, CQT and CN samples. This effect is clearer in the samples

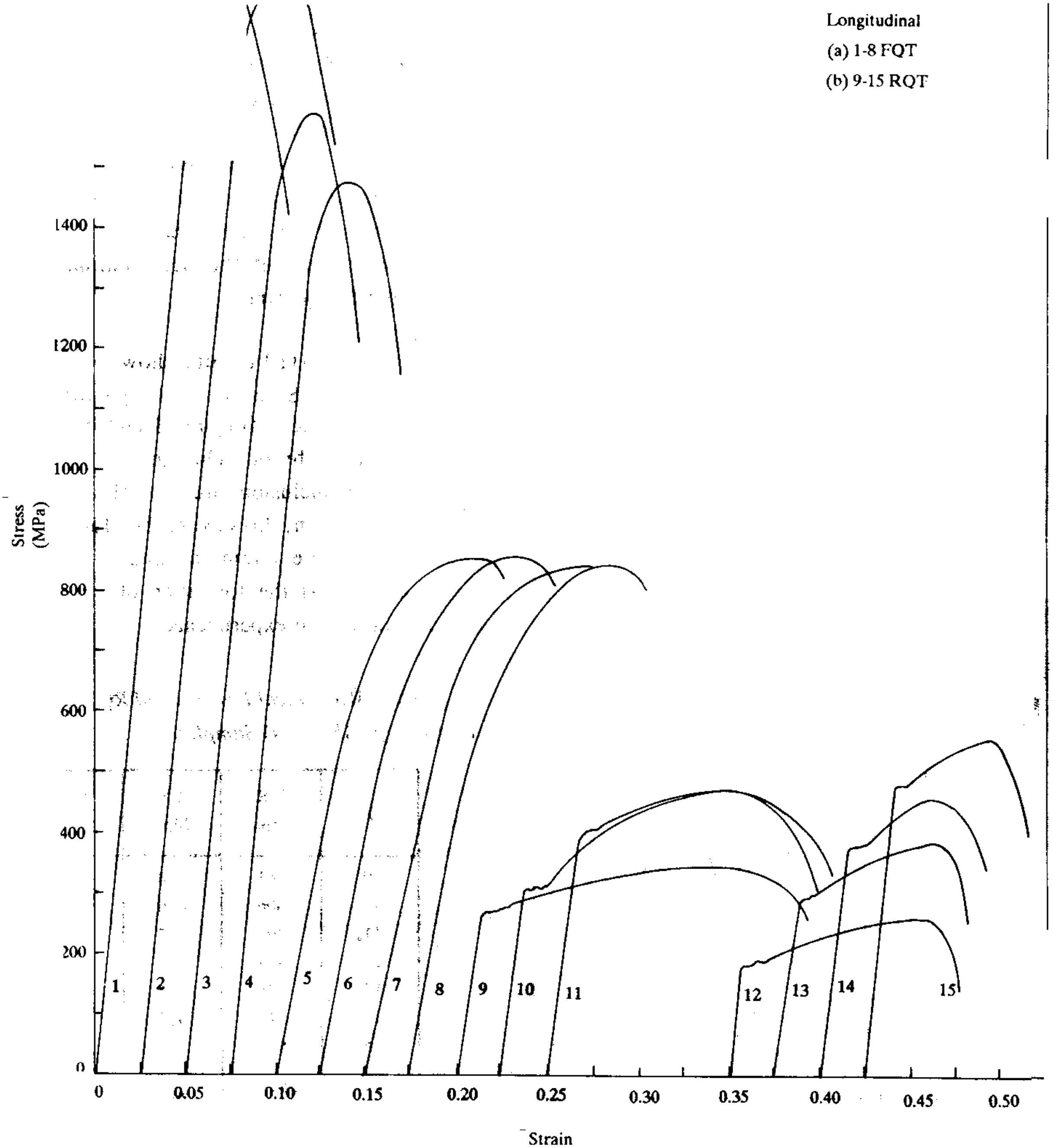


Figure 3. Stress-strain curves in longitudinal direction for (a) FQT samples 1 to 8 and (b) RQT samples 9 to 15.

with lower amounts of alloying elements.

Deformation processing in prior austenite can also influence the gamma to alpha transformation temperature by extending the ferrite range to higher temperatures. This is usually due to strain-induced transport of C and alloying elements to austenite which shifts the formation of bainite to lower temperatures.

The effects of the microstructural change due to nonisothermal rolling on hardenability of the samples are shown in the upper-left of Figure 2. The diminution of hardenability of the samples versus reduction ratio can be due to both the increase in the size of the precipitates and the variation of the gamma to alpha transformation temperature [9].

Nb helps austenite to ferrite transformation by blocking carbon and thus forming carbide nucleation centers which enhance the rate of transformation. V also has an increasing effect on the rate of transformation but to a lesser extent. These effects accompanied by the pinning of the dislocations can result in a very fine ferrite structure, provided that the microalloying contents do not exceed a certain limit (Figure 1). Choosing a lower rolling-temperature suppresses precipitation in austenite and preserves microalloying potential for precipitation in the ferrite which is dispersed finely and more effectively for strengthening effects. Precipitation is thus controlled as a decisive process during simultaneous rolling and cooling schedules. The rate of precipitation depends on the temperature range through which the rolling is accomplished, the supersaturation of the alloying elements and the deformation in the austenite and ferrite range.

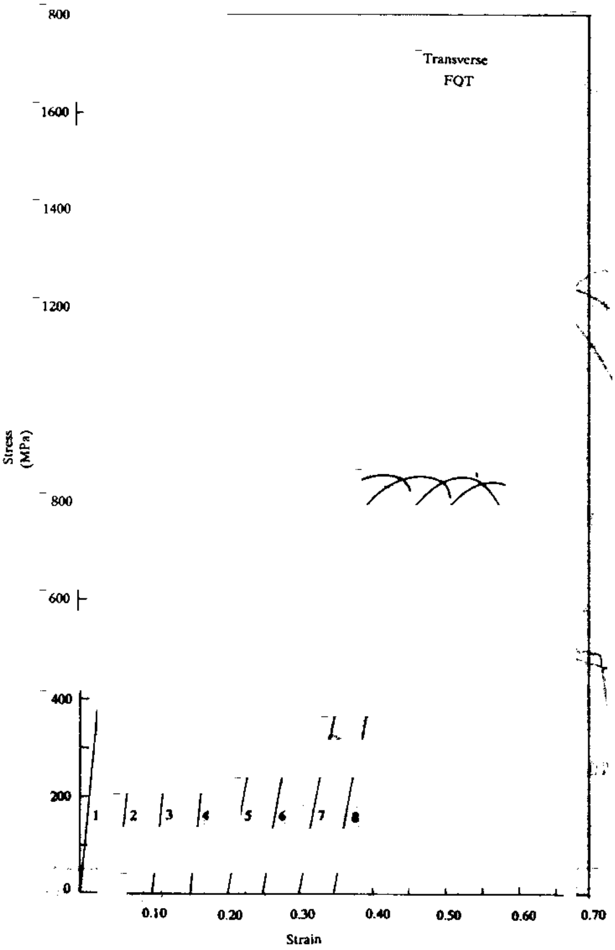


Figure4. Stress-strain curves in transverse direction for FQT samples 1 to 8

REM Effects

The experimental results show a decrease in Y. S., U. T. S. and % elongation from sample 1 to sample 4 (Figures 3 and 4). This is in spite of the obvious change in shape and analysis of the inclusions due to REM additon. These results can, however, be due to the differences in the chemical analysis of the samples as well as the formation of oxy-sulfide inclusions during the experiments.

Table 3. Mechanical Properties of RQT (9 to 15) And FQT (1 And 4) Samples.

Sample	Y.S. Mpa	T.S. Mpa	El.in 50mm,%
9	261	350	24.4
10	306	406	22.6
11	406	477	19.6
12	375	448	14.9
13	438	513	14.1
14	543	625	13.9
15	680	766	13.6
1	1483L 1498T	1795L	11.1L 3.9T
4	1272L 1218T	1494L	107L 1.2T

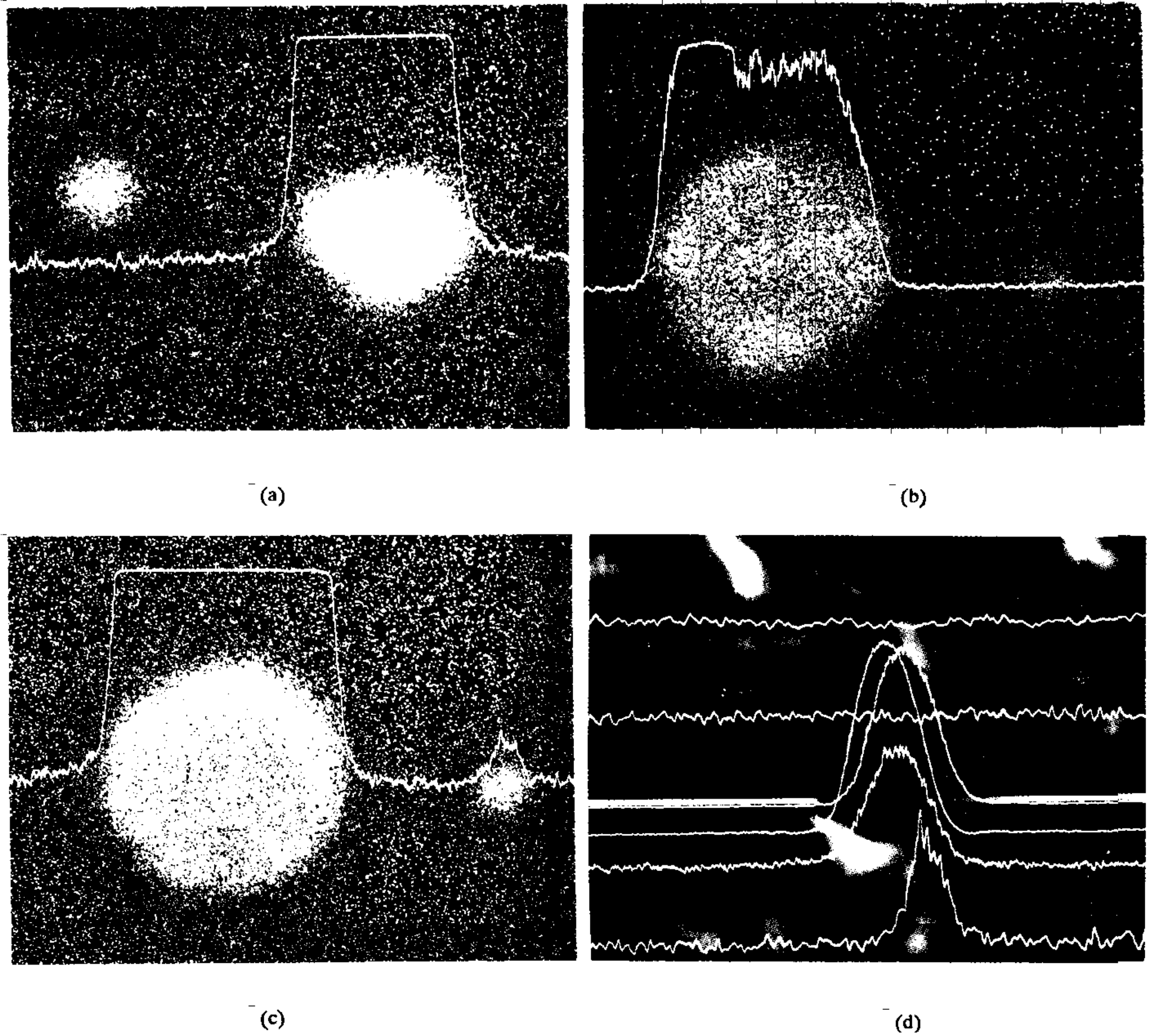
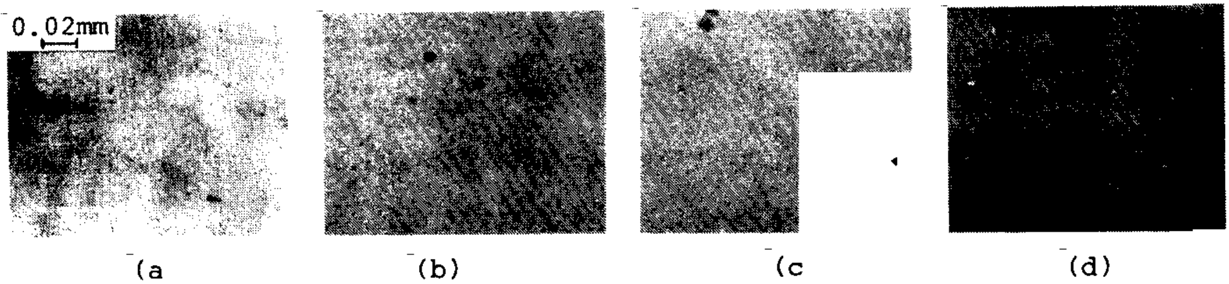

(a) (b) (c) (d)

Figure 5. Dark-field electron micrographs of precipitates in (a) CQT, (b) RQT, (c) FQT and (d) CQT specimens. Lines show typical microanalyses of elements in the samples: (a) V, (b) Nb, (c) Ce and (d) lines from bottom to the top Nb, V, Mn, S,P and Ca. The magnification is X4000.



(a (b) (c (d)

Figure 6. Optical micrographs of inclusions in (a) FQT without REM (Longitudinal), (b) FQT with REM (Longitudinal), (c) FQT without REM (Transverse) and (d) FQT with REM (Transverse).

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