DISTORTION AFTER VACUUM HEAT TREATMENT OF AISI D2 , 8%CR 2%MO 1.6%V AND A 7.5%CR 1.3%MO 2.7%V (P/M), COLD WORK TOOL STEELS[1]

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Abstract

The distortion after vacuum heat treatment has been investigated for three cold work tool steels: a traditional 1.5% C 12%Cr 1% Mo 1%V, AISI D2 steel; a recently developed 1.0%C 8%Cr 2 %Mo 1.6%V steel and a 0.8%C 7.5%Cr 1.3%Mo 2.75%V (P/M) steel. The degree of distortion was assessed via out-of-roundness measurements, using a tri-dimensional measuring machine .The results showed that the shape and magnitude of distortion was different for each steel along the heat treatment stages and it is associated with the composition as well as the microstructural differences of each steel. The size, volume fraction and distribution of primary carbides, together the retained austenite content, all played important roles on the distortion after heat treatment under vacuum of the investigated steels. The smallest distortion were observed with P/M steel, followed closely by the 8%Cr steel and finally, by the conventional D2 steel. The smallest and more uniform distortion of the P/M steel has been attributed to its smaller, spherically shaped and better distributed primary carbides when compared with the both conventionally cast 8% or 12% Cr tool steels..

Key words: Distortion; P/M; Tool steels.

DISTORÇÃO APÓS TRATAMENTO TÉRMICO À VACUO EM AÇOS FERRAMENTAS PARA TRABALHO A FRIO: AISI D2; 8%CR 2% MO1.6%V ; 7.5%CR 1.3%MO E 2.7%V (P/M)

Resumo

A distorção após tratamento térmico a vácuo foi investigada para três aços ferramentas para trabalho a frio: o tradicional aço AISI D2 (~1.5%C 12%Cr 1% Mo) ; um aço recentemente desenvolvido (~1%C 8% Cr 2% Mo 1% V) ; um aço produzido por metalurgia do pó (P/M), (~ 0.8%¨ C 7.5%Cr 1.3%Mo 2.75% V) . A distorção foi avaliada através de medidas de não-circularidade com um máquina de coordenadas tri-dimensional. Os resultados mostraram que a forma e magnitude da distorção foi diferente para aço ao longo dos estágios de tempera e revenimento, e foi associada com a composição e microestrutura de cada aço. O tamanho, fração volumétrica e distribuição de carbonetos primários juntamente com o teor de austenita retida tiveram influência nas distorções abservadas. A menor distorção foi observada com o aço P/M, seguida pelo aço com 8%Cr e finalmente o aço AISI D2 que apresentou a maior distorção. A menor e mais uniforme distorção apresentada pelo aço P/M foi atribuida a presença de carbonetos primários mais esféricos, menores e mais uniformente distribuidos com comparado com os outros aços.

Palavras-chave: Distorção: Metalurgia do pó; Aços ferramenta.

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INTRODUCTION

The AISI D2, or DIN WNr.1.2379 (≈1. 55%C 12%Cr 0.9%Mo 0.9%V) tool steel, is still one of the most popular grades for cold work applications requiring high wear resistance. Nevertheless, there are some serious drawbacks associated with this traditional high carbon, high chromium, ledeburitic steel such as its moderate toughness, low machinability and proneness to distortion after heat treatment. The later becomes critical in many applications, particularly for tools of intricate, complex or high precision such as in industrial flat or circular knives, thread rolling dies, gages, punches, precision dies, etc. Nevertheless, a limited number of research works are available in the literature on the distortion of ledeburitic steels, even though they are used in virtually all manufacturing processes.
More recently, a new generation of 1%C - 8%Cr based cold work steels has been developed[1,2] and due to their better balance of carbon and carbide forming elements, better homogeneity, size and distribution of second phase particles improved re-melting techniques such ESR (electro-slag refining) or VAR (vacuum arc refining) processes, has led to a cold work steel with better toughness than the original D2 steels. An alternative route to improve even further the microstructure of tool steels is via powder metallurgy (P/M), where a dramatic increase in the solidification rate causes the formation of very fine microstructure and second phase particles, leading to better toughness, heat treatment response and nearly isotropic properties.[3-4]
This work is aimed at investigating the distortion, during vacuum heat treatment of the traditional AISI D2, a recently developed ledeburitic 1.0%C - 8%Cr – 2 %Mo 1.6%V steel and a 0.8%C - 7.5% Cr - 1.3% Mo - 2.75%V, rapidly solidified, P/M , cold work tool steels.

EXPERIMENTAL PROCEDURE: MATERIALS AND METHODS

Samples, 40 mm round x 80 mm long, standard hot rolled and annealed, were used in this investigation. The chemical analysis carried out with the three investigated steels might be seen in Table 1 :

Table 1. Chemical Composition (weight %) of the steels investigated

Steel	Denomination	%C	%Mn	%Si	%S	%Cr	%Mo	%V
A	D2	1.53	0.33	0.40	0.007	11.59	0.65	1.17
B	8%Cr	1.00	0.34	0.90	0.007	7.87	1.61	1.86
C	P/M 3V	0.80	0.38	0.96	0.013	7.70	1.40	2.84

Steel A shows the classic composition of a D2, ledeburitic steel, with its high Cr and C and about 1% V, contents, whereas steel B has much less carbon and Cr and a higher Mo and a slightly higher vanadium contents than A. On the other hand, the P/M steel has a much higher vanadium and a slightly lower molybdenum contents than steel B, with the lowest carbon content of all three steels. The content of Mn and Si are nearly the same for all steels. All three samples have been previous machined and centerless finish ground to the dimensions of 40,000 -0/+0.001 round x 80 mm.
A 10 bar Ipsen vacuum furnace from Brasimet's facilities in São Paulo, Brazil, has been used as heat treating media for all 03 steels. The samples have been loaded together, at the same time and at the furnace position. The temperature and time

were continuously computer monitored. Three pre-heatings were used: at 600, 850 and 980ºC, with holding times of 15, 39 and 17 minutes, respectively. When the temperature reached 1030ºC, the samples were held for 36 minutes after which a 10 atm N_2 gas quenching was applied . After which, 03 tempers of 02 hours each were carried out at temperature of 540ºC.
The dimensional measurements before the heat treatment, after quenching and the three tempers stages, have been all carried out in a Mitutoyo's QM Mercure 333, three-dimensional measuring machine, with a five ruby tip. The measurements have been concentrated along the external diameter of the samples, at the longitudinal (Z) positions of 10, 15 and 20 mm from bottom (0 mm) of the surface of the samples which was in permanent contact with the surface of the metal basket (used to keep the samples inside the vacuum chamber). Three measurements were taken at every 15^o along the diameter of the samples so that 24 diameter measurements were collected for the data analysis. All measurements have been carried out at constant temperature of 21^oC and before that a calibration procedure was used with a standard 25 mm sphere. The accuracy yielded by the equipment is +/- 0.5 μm.
Backscattered (BS) and secondary electron (SE) images together EDS and/or WDS microprobe microanalysis facilities, have been the main technique together optical microscopy as a supporting technique, to analyze the microstructure of the as-received (annealed) and heat-treated samples. Optical microscopy and hardness tests were carried out at the Metallurgical Center of Brasimet, São Paulo, Brazil whereas the electron microscopy analysis was conducted at both, Faculty of Geosciences and Mining & Petroleum Engineering Department, Politecnica School of Engineering, São Paulo University (USP), São Paulo, Brazil.

3 RESULTS AND DISCUSSION

The diameter evolution of the three steels, before heat treatment, after quenching and after the first, second and third tempers, are shown in Figure 1. The results show that all three steels increased the diameter after the final temper: from the original 40,000 mm to 40.040 mm for the 8%Cr steel, to about 40,050 mm for the P/M 3V steel and to about 40,055mm for the D2 steel. The magnitude of diameter change in percentage, along the heat treatment for all steels is displayed in Figure 2 which shows that the D2 and P/M 3V steels increased their final diameters in about 0.13 % whereas the 8%Cr steel increased slightly less, i.e., 0.11%.
Polar charts have been used to assess the out-of-roundness of all three steels along the heat treatment process. However, due to the significant diameter differences shown by the three steels along the heat treatment cycle, polar charts had to be separated in two axis scales to decrease data pollution and to improve visualization and details: the as-annealed and as-quenched diameters, as well as the theoretical diameter of 40.000 mm are all shown on the left of Figure 3, whereas the diameter as a function of the three tempers are shown on the right of the same figure. The amplitude of distortion, evaluated from the difference between the smallest to the largest diameter, was found to be 0.003 mm (3 μm) for the P/M steel, 0.010 mm (10 μm) for the 8%Cr steel and finally, 0.015 mm (15 μm) for the D2 steel. As far as the shape of distortion is concerned, the P/M 3V steel presented a more uniform shape not only after the final temper stage but even after it was quenched, followed by the 8%Cr steel and finally by the D2 steel.
Typical backscattered (BS) and secondary electron (SE) images of all steels before heat treating, i.e., in the as-annealed condition, after quenching and after the third

temper are shown on Figure 4, at various magnifications. The as-annealed microstructures of all steels show quite distinct features, regarding sizes, distribution, morphologies as well as chemistry of the particles reflected by the different grey, black and white colours of the carbides which have been qualitatively identified via energy dispersive spectroscopy analysis (EDS). As the production route and chemistry of D2 and 8%Cr steels are more closely related each other than the P/M 3V steel, a comparison between the two conventional steels will be dealt first.

The as-annealed microstructures of D2 and 8%Cr steels present quite distinct features, regarding sizes, distribution, morphologies and chemistry of second phase particles, as view via BS and SE images. For instance, in the D2 steel, it is observed the presence of large and grey coloured particles which are irregular shaped and of large size, varying from about 20 μm in diameter for the more circular ones and up to 5 μm wide and 30 μm long for the needle shaped ones. Via EDS microanalysis, these blocky particles have been identified as Cr-Fe-Mo-V-complex carbides with a typical weight composition of: C=12.9%; Cr =39.97%; V=5.69% and Mo=3.18%. An attempt was made to evaluate their volume fraction via SEM + EDS and the average value found for 05 fields was 14.7%. It was also observed in the annealed D2 steel the presence of much smaller (about 2 μm= 2000 A) rounded carbides which are mostly grey but also, some other lighter grey coloured and even smaller (800 to 1000A) particles. Via EDS analysis, the former were identified as Cr-Fe-Mo-V complex carbides with however, a higher Mo content than the blocky carbides. A typical weight composition of these carbides was: C=11.05; Cr=34.06%; Fe=46.64%; Mo=6.85%; V=0.96%

The as-annealed microstructure of the 8%Cr steel, instead of massive and more angular Cr carbides, it is observed the presence of much smaller (about 10 μm), dark grey and more rounded particles; a large volume fraction of very small, grey, rounded particles (about 1-2 μm), spread all over the matrix, together the presence of also very small white rounded particles (about 1 μm). Finally, black particles, platelet (about 10 μm long and 2 μm wide) shaped in smaller quantity together a large volume fraction of very small round ones (about 1 μm which at higher magnification revealed to have a grey colour at their external areas). Via EDS microanalysis, the larger grey, white and black particles were identified as follows: grey particles: Cr-Fe-V-Mo complex carbides with a typical weight composition of: C=11.17%; Cr=36.11%; Fe=38.27%; V=10.39%; Mo =4.05%; black particles: typical VC carbide with some Mo dissolved; white particles: Mo-Fe-Cr-V complex carbides with C=9.64%; Mo= 44.80%; Fe=34.16%; Cr=4.68%; V=3.06%.

Via BS images, it was possible to identify the presence of retained austenite in both steels although with the D2 steel, it was more easily observed than with 8%Cr steel, indicating thus, a higher volume fraction with the conventional D2 steel. Via BS and EDS, a volume fraction micrographs of retained austenite of 9.3% was found. Via X-ray diffractometry technique, the results found were 19.9 % and 14.3 % for the D2 and 8%Cr steels, respectively. After the first temper, the level dropped to values of 1-2%, which is below the necessary accuracy to use the x-ray diffractometer for this purpose.

The microstructures displayed via BS and SE images of both steels, immediately after quenching from the austenitizing temperature of 1030^{o}C revealed clearly a martensitic matrix. No significant differences via SE image were found between the steels which is bound to be found with other higher resolution electron microscopy such as TEM. Nevertheless, with the D2 steel, it was seen a higher volume fraction of small (< 1000A) round carbides which have been preferentially precipitated along the austenite grain boundaries as well over the matrix. Comparing with the as-annealed microstructure, it was noted that the lighter grey carbides have disappeared and new, grey coloured,

more rounded and most importantly, smaller (800A to 1000A) particles has appeared in the microstructure. These new particles are pro-eutectoid carbides which have precipitated during the cooling.

After the third temper, the matrix of both steels are clearly tempered martensite. The measured hardness of all steels was as within the range 56-58 HRC. As far as the carbides are concerned, new and very small carbides (about 400-600°A) were observed at higher magnifications for the D2 steel, whereas for 8%Cr, particles even smaller (200-300A) and at higher volume fraction was seen with this steel. Such differences regarding carbides volume fraction and size indicate that more abundant secondary precipitation occurred after the third temper with the 8%Cr steel. Nevertheless, their presence does not appear to contribute dramatically to the distortion of 8%Cr steel.

Thus, the higher volume of large primary carbides, higher carbon and chromium contents together its higher retained austenite volume fraction all appear to play more important role to explain the worse distortion of D2 when compared with 8%Cr steel. The latter, with its higher V and lower C contents, guarantees that more carbon is tied with vanadium forming the stable MC carbide type. As a result, much less carbon is left into solution in the matrix to induce the presence of retained austenite, so that the distortion is smaller.

The as-annealed microstructure of the P/M 3V steel revealed that the carbide size and distribution of this P/M steel is markedly different than the other two conventionally ingot cast steels. The carbides are much smaller in size and homogeneously distributed throughout the matrix. The electron images revealed the presence of small, platelet shaped and white carbides. EDS microanalysis revealed that the small (about 2-4 μm), normally black, rounded particles, are typical vanadium MC type carbide with some Mo dissolved, and are homogeneously distributed throughout the matrix. The white particles were identified as complex Fe-Cr-Mo-V carbides. These latter particles appears to be preferentially formed along the original austenite grains indicating that they might have been precipitated along the annealing or preliminary hot working production stages of this steel which includes, after hot isostatic pressing, the usual forging and hot rolled steps to reach the final gage. After quenching, the complex Fe-Cr-Mo-V carbides, found in the as-annealed microstructure, have disappeared. X-ray diffractometry measurements showed a retained austenite content of 4.3%. After the third tempering no significant differences regarding carbides volume fraction have been found when compared with the as-quenched microstructure. In the case of the P/M 3V, due to its lower carbon and higher vanadium than the 8%Cr steel, more MC carbide is produced so that even lesser carbon is left into austenite generating, thus, a lower volume fraction of austenite after quenching.

Thus, the best distortion results showed by the P/M 3V steel might be explained by its better balanced composition together its finer microstructure constituted of very small, rounded and more homogeneously distributed VC carbides which are originated from the rapid solidification process. The 8%Cr steel, despite its higher retained austenite content than the P/M steel, has smaller carbides than the traditional D2 steel this explains its good distortion results. Finally, the D2, with its high volume fraction of massive and irregular chromium carbides, together its high retained austenite after quenching, explains its poor distortion results. Although more work is currently under progress, the preliminary results of this work have demonstrated that the fine microstructural differences, inherent to each one of the investigated cold work steels and which can be only revealed by high resolution

electron microscopy, have shown to contribute markedly to the final distortion of the steels.
Finally, the difference in shape showed by the various steels indicates that the technique employed in this work which includes the measuring equipment, size samples, sample furnace conditioning, surface finish conditions, etc, has been sensitive enough to monitor the dimensional changes taking place inside each sample so that the differences found must be related to material, chemistry and phase transformations which are inherent to each steel, following the heat treatment media and procedure chosen.

4 CONCLUSIONS

1. The distortion during vacuum heat treatment for a D2, a 8%Cr and a P/M 3V cold work steels have been compared in the laboratory;
2. The dimensional results obtained agreed with the literature demonstrating that the technique employed, using a three dimensional measuring machine, was sensitive enough to assess true material properties differences affecting the distortion of the steels;
3. The least distortion was shown by the P/M 3V steel, followed closely by the 8%Cr steel and finally by the traditional D2 steel;
4. The microstructural analysis revealed that secondary carbides and retained austenite played important role on the distortion of the investigated steels;
5. The best overall results obtained with the P/M steel has been attributed to the presence of small, round and stable primary VC primary carbides, homogeneously distributed throughout the matrix.

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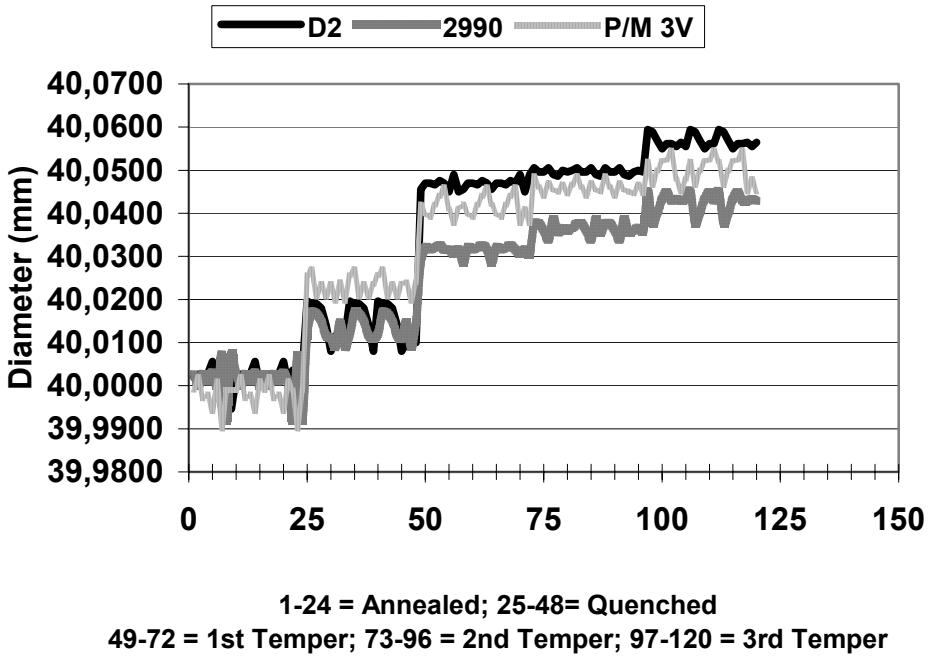

Figure 1. Diameter evolution for the D2, 8%Cr and P/M 3V steels, before the heat treatment, after quenching and the three subsequent tempers

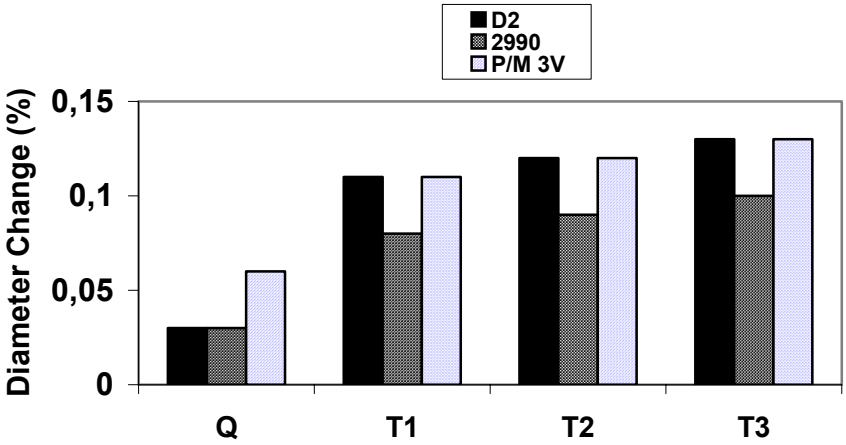

Figure 2. The percentage diameter change for D2, 8%Cr and P/M 3V steels after quenching and the three subsequent

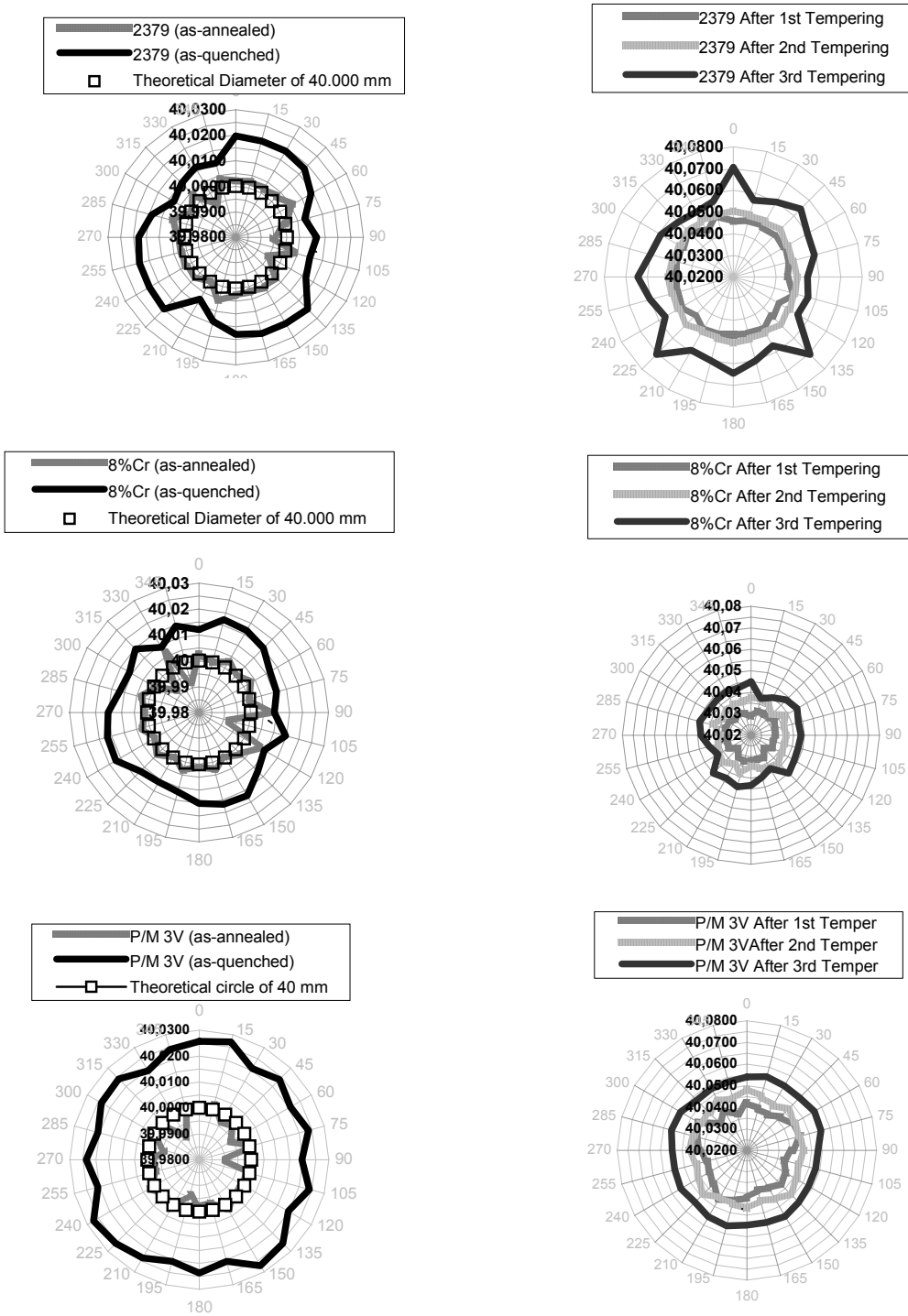

Figure 3. Out-of-roundness polar charts for the D2, 8%Cr and P/M 3V steels: on the left = before heat treatment (as-annealed) & after quenching; at the right = after the first, second and third tempers.

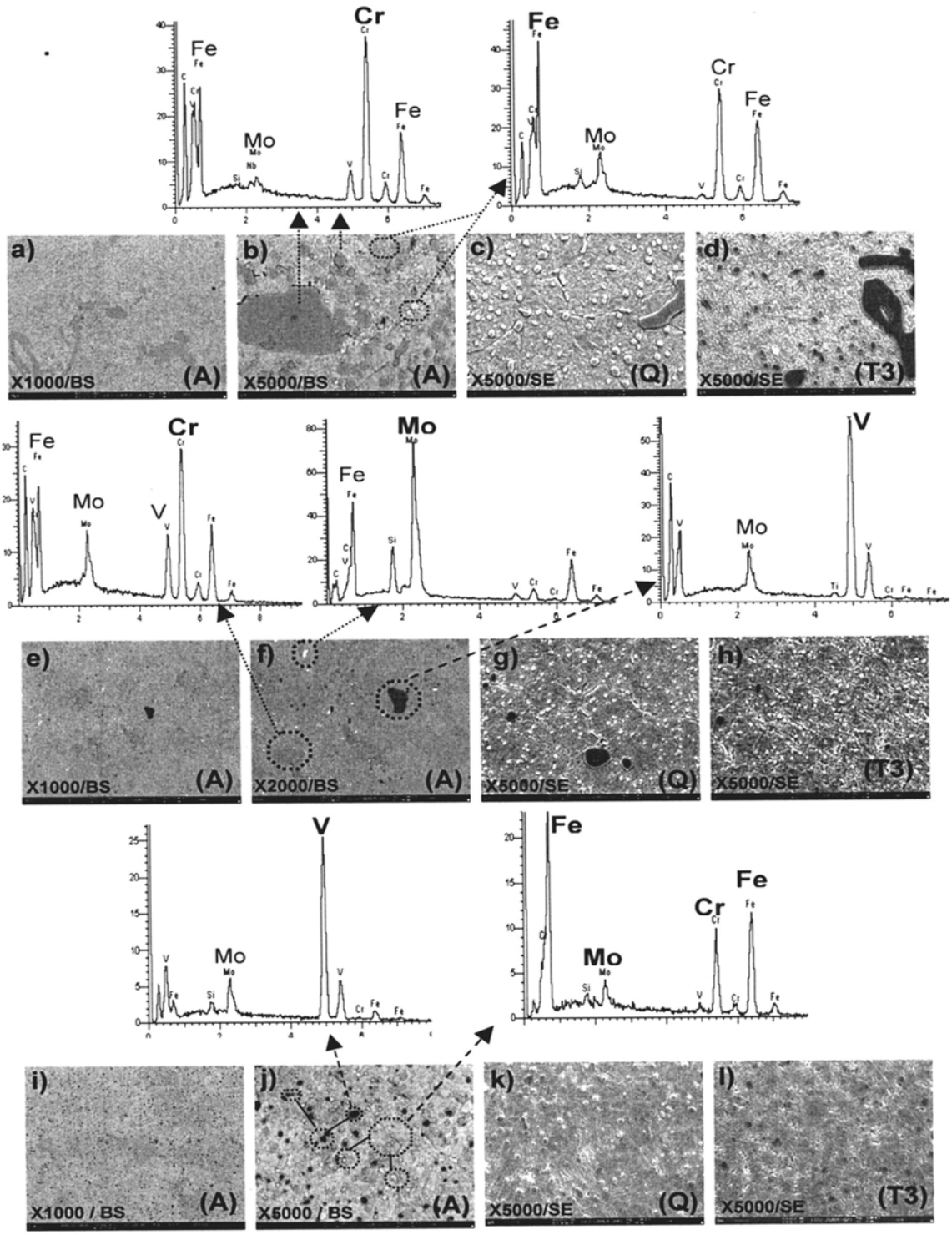

Figure 4. SE/BS images of the **D2** (**a-d**), **8%Cr** (**e-h**) and **P/M 3V** (**i-l**), steels, in the: **(A)= as-annealed** (+ EDS analysis), (**Q**) = **as-quenched**; (**T3**)= **3rd temper** .