ISSN 1516-392X

MORPHOLOGICAL TRANSITION OF MARTENSITE AS RELATED TO Ni CONTENT IN A Fe-5%Ni/Fe-10%Ni DIFFUSION COUPLE[1]

Eduardo Franco de Monlevade[2]
Carlos Eduardo Pinedo[3]
Hélio Goldenstein[4]

Abstract

A diffusion couple composed of Fe-5%Ni and Fe-10%Ni alloys was carburized in a salt bath to 0.8%C through the entire thickness. The samples had a smooth Ni gradient ranging from 5 to 10% Ni. Samples were carburized at 900°C, and cooled in oil. A morphological transition in martensite and an increase in the volume fraction of retained austenite are clearly visible as the Nickel content rises towards 10%. Grain boundary formation of bainite is also visible in the lower Nickel content regions. The formation of bainita ceases at an approximate Nickel content of 6.5%, and the scale transition of martensite (from very fine plate to large plate morphology) is more pronounced shortly afterwards, at approximately 7% Ni. An increase in the amount of retained austenite is also present as the nickel content increases, and the beginning of its appearance seems to be closely related to the change in martensite morphology.
Keywords: Martensite; Morphological transitions; Austenite decomposition.

TRANSIÇÃO MORFOLÓGICA DA MARTENSITA EM FUNÇÃO DO TEOR DE NÍQUEL EM UM PAR DE DIFUSÃO Fe-5%Ni/Fe-10%Ni

Resumo

Um par de difusão composto de ligas Fe-5%Ni e Fe-10%Ni foi carbonetado em banho de sal para um teor de carbono de 0,8%C ao longo da espessura. As amostras tinham um gradiente de composição entre 5% e 10% Ni. A carbonetação foi feita a 900°C, seguida de resfriamento em óleo. Uma transição morfológica da martensita e um aumento da fração de austenita retida são claramente observáveis à medida em que o teor de níquel sobe até 10%. A formação de bainita a partir dos contornos de grão também é visível nas regiões de baixo níquel. A formação de bainita cessa em um teor de níquel de aproximadamente 6,5% , e a transição na escala da martensita é mais pronunciada em teores pouco maiores, de cerca de 7%. Um aumento na quantidade de austenita retida também se observa com o aumento do teor de níquel, e o começo do aparecimento da austenita retida parece estar intimamente ligado à mudança na escala da martensita.
Palavras-chave: Martensita; Transições morfológicas; Decomposição da austenita.

[1] *Technical contribution to 66h ABM Annual Congress, July, 18th to 22th, 2011, São Paulo, SP, Brazil.*
[2] *Research and Development Specialist, Mangels Steel Division.; PhD student, Escola Politécnica da Universidade de São Paulo – Dept. of Metallurgical and Materials Engineering*
[3] *Professor, Universidade de Moji das Cruzes*
[4] *Professor, Escola Politécnica da Universidade de São Paulo – Dept. of Metallurgical and Materials Engineering*

1 INTRODUCTION

It is a very well known and accepted fact that alloying elements have a crucial role in determining the hardenability of steels. The addition of certain elements to steel can stabilize austenite, and one of the visible effects of this stabilization is the change in the morphology and amount of martensite formed after fast cooling from a given temperature. Among these austenite stabilizing elements, nickel has always been one of the most popular choices for scientists. Several authors,[1-3] dedicated time to studying the Fe-Ni or Fe-Ni-C system, due to nickel's very reduced tendency to form carbides, added to its tendency of slowing down carbon diffusion. Added to this, the Fe-Ni-C system is very suitable to study the martensitic transformation, as it allows for obtaining mostly all possible morphologies of martensite as well as large quantities of retained austenite for higher nickel contents, which allows for precise crystallographic studies.
One very effective way to study transitions in behavior regarding phase transitions is the use of diffusion couples. This method was described by Hutchinson et al.,[4] and has the great advantage of eliminating the need for production of several different alloys to study the influence of a certain alloying element. Examples of studies using this method are studies by Sinclair et al.[5] on the effects of niobium on recrystallization kinetics and by Hutchinson et al.[6] on the effect of nickel on allotriomorphic ferrite formation kinetics. Using diffusion couples, one can identify critical or particular transitions in behavior, and use that as a basis for alloy design, or cast an alloy of specific composition based on results obtained with the diffusion couple. This allows for considerable savings in alloy design, or in studies of the effect of alloying elements in the behavior of materials, as the need to cast several different alloys is eliminated.

2 EXPERIMENTAL PROCEDURES

Diffusion couples were produced from a Fe-5%Ni and a Fe-10%NI alloys by hot cladding. The couples were produced from cast and hot forged bars. The as received bars were machined to remove the oxide layer from both surfaces. One face from each machined block was further rectified. The rectified surfaces were put in contact and the two machined blocks were welded together. The whole set was then heated to 1200°C and hot rolled. The result is a bulk diffusion couple, about 450 mm in length and 20 mm in thickness. The several steps for production of the diffusion couples are shown in Figures 1 and 2. The diffusion couple was produced at Serras Saturno, in São Paulo, using a procedure developed in the 1960s by *Alpont S/A Produtos Siderurgicos,* for the production of knives for steel cutting. The chemical compositions of the two alloys used to produce the diffusion couples are given in Table 1. The finished diffusion couple is shown in Figure 3.

Table 1 - Chemical composition of the Fe-5%Ni and Fe-10%Ni used to produce the diffusion couple

Fe-Ni-5	***C***	***Ni***	***Si***	***Mn***	***Cr***	***Mo***	***W***	***V***
	0,010	*4,91*	*0,030*	*0,030*	*0,090*	*0,040*	*<0,010*	*<0,010*
	Ti	***Nb***	***Co***	***Cu***	***Al***	***P***	***S***	***N_2***
	<0,0050	*<0,010*	*0,010*	*0,050*	*<0,0050*	*<0,0050*	*0,001*	*0,0048*
Fe-Ni-10	***C***	***Ni***	***Si***	***Mn***	***Cr***	***Mo***	***W***	***V***
	0,003	*9,94*	*0,020*	*<0,010*	*0,090*	*0,040*	*0,030*	*<0,010*
	Ti	***Nb***	***Co***	***Cu***	***Al***	***P***	***S***	***N_2***
	<0,0050	*<0,010*	*0,010*	*0,050*	*<0,0500*	*<0,0050*	*0,001*	*0,0042*

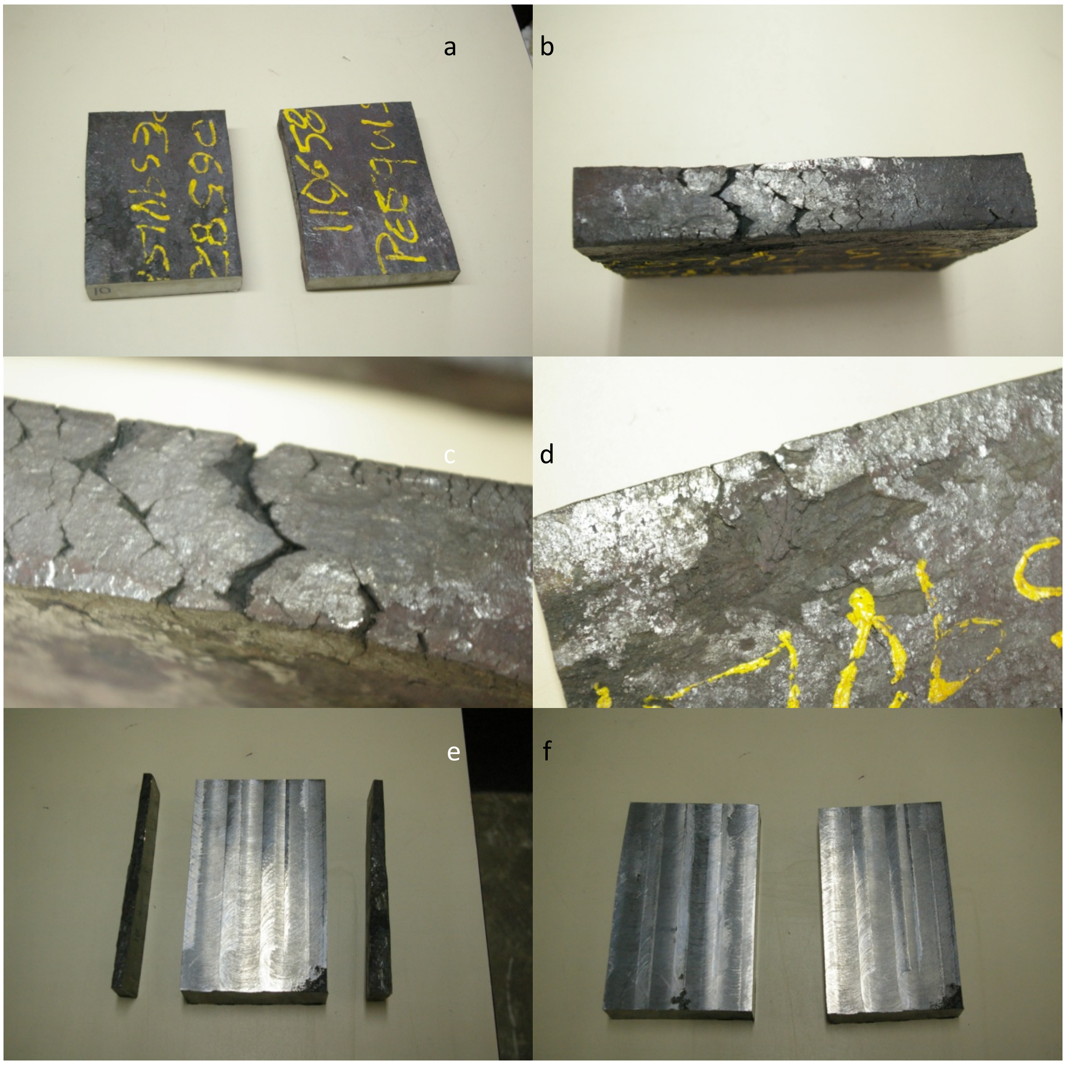

Figure 1 - Preparation steps for production of the diffusion couples; a) separation of blocks from the original forged bars; b, c and d) close view of the edge cracks and deep oxide layers; e) edges of the blocks sectioned away to avoid edge cracks; f) machined blocks.

The resulting diffusion couple had no problems with oxide layers between the two alloys. The interface between the 10%Ni and the 5%Ni regions is shown in Figure 4.

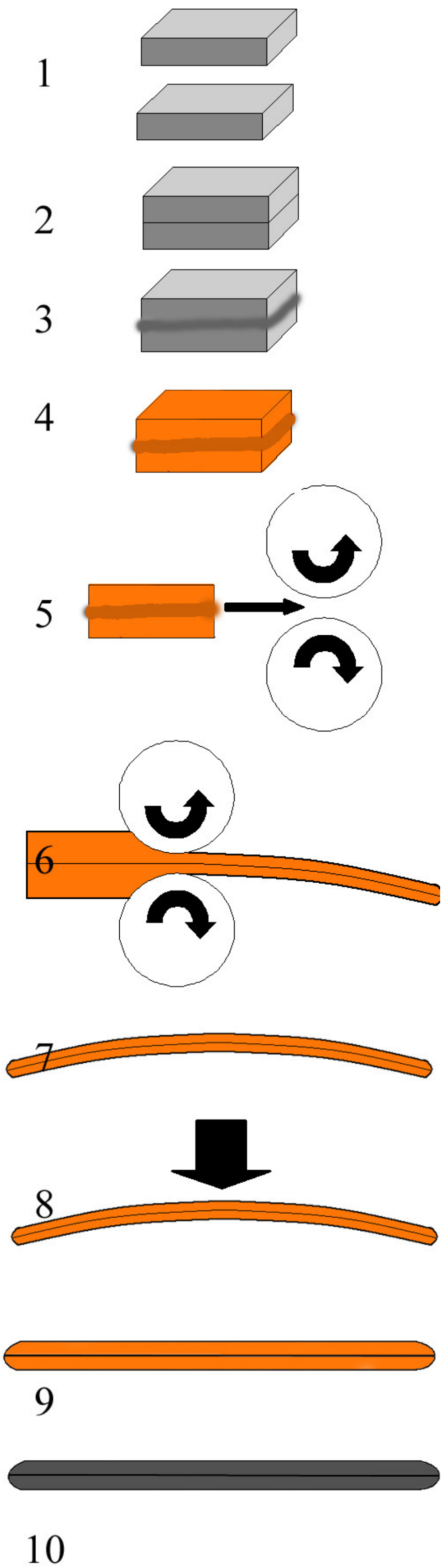

Figure 2 - Processing sequence of the diffusion couple.

ISSN 1516-392X

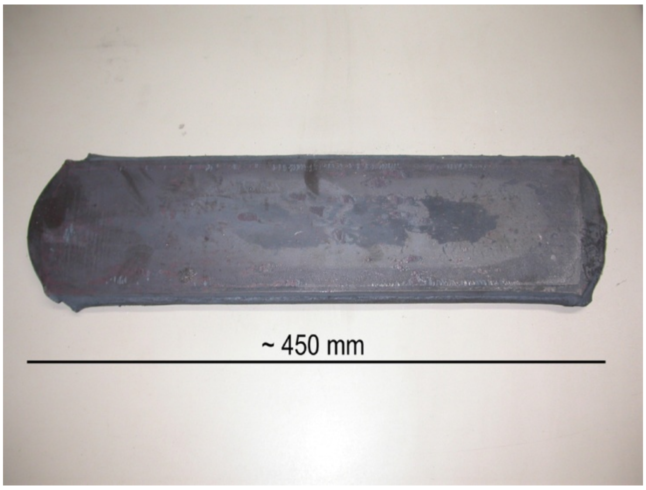

Figure 3 - finished diffusion couple.

The couples were solution treated at 1350°C for 36 hours to allow for the formation of the Ni gradient. The resulting Nickel gradient is about 0.3 mm thick. In order to extend this region, the couples were cold rolled, with the Ni gradient aligned with the rolling direction, with a 90% reduction, thus extending the nickel gradient by a factor of 10. After rolling, the couples were treated at 1300°C for 24 hours to eliminate heterogeneities originated from the cold rolling process. The diffusion couples were then carburized in an industrial salt bath to a carbon content of 0.8% through the entire thickness. The solution treatment was carried out in a sealed quartz tube, under vacuum, and the carburized sample was quenched in oil from a temperature of approximately 900°C. The sample was then submitted to standard metallographic sample preparation procedure, and etched with 3% Nital. The sample was observed in the optical microscope and submitted to scanning electron microscopy and Energy Dispersion X-Ray (EDS) analyses for determination of nickel contents at which transition might take place.

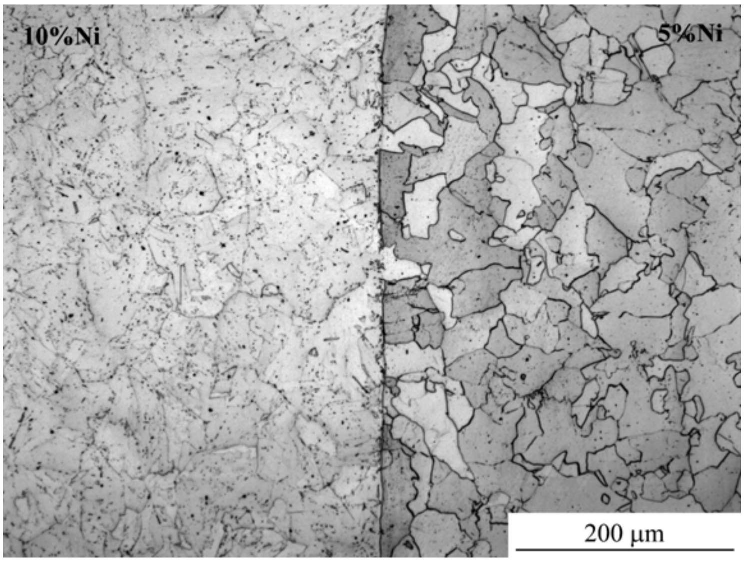

Figure 4 – Microstructure of the interface region in the as rolled diffusion couple.

3 RESULTS AND DISCUSSION

An overlay of the gradient region with the nickel content determined by EDS analysis is shown in Figure 5. It is possible to observe that there is the formation of eutectoid decomposition products on the lower nickel side of the couple. The structure is fully martensitic after the Ni content reaches approximately 6.5%. The gradient region spans a length of about 4 mm.

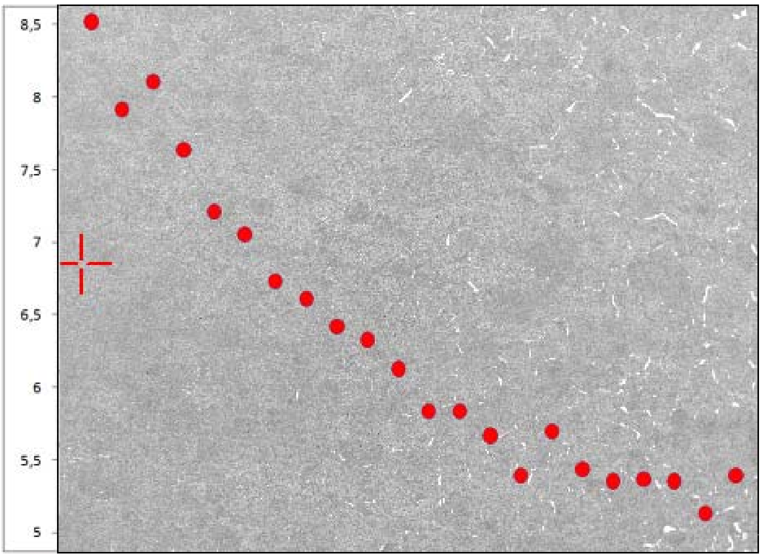

Figure 5 - Overlay of the gradient region and the Ni content determined by EDS analysis.

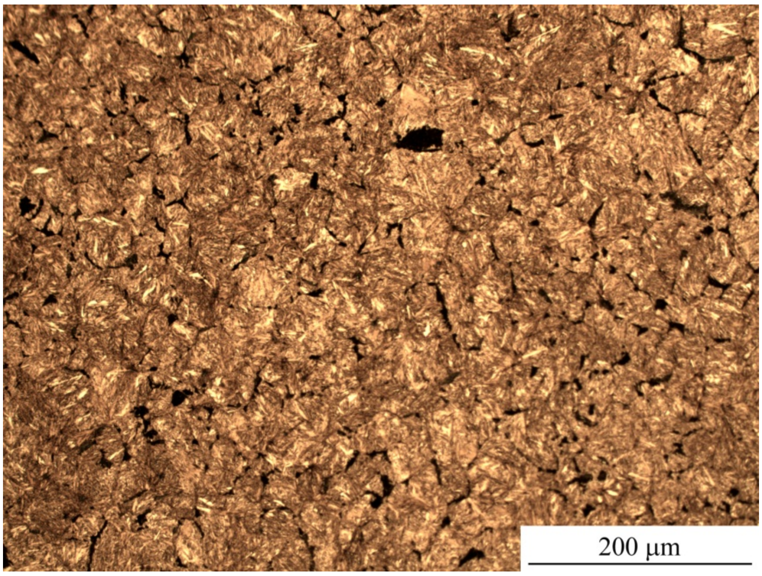

Figure 6 - Low Ni region of the diffusion couple.

Optical microscopy images of selected regions of the diffusion couple are shown in Figures 6-8. It is possible to see that the martensite morphology appears to be of very fine plate type at low nickel regions. As the nickel content rises, the martensite morphology switches to plate, and the quantity of retained austenite increases dramatically. The increase of retained austenite is more pronounced after the cessation of the eutectoid decomposition products. A higher magnification view of the martensite at 5%Ni is given in Figure 9, and a high magnification view of the martensite at 10%Ni region is given in Figure 10. The high magnification images

ISSN 1516-392X

allow for the observation of a very fine plate type martensite formed at low nickel regions, with even finer martensite plates filling the spaces between the larger plates. On the other hand, on the high nickel region, the martensite plates are large, and the space between plates was only slightly filled with new, smaller martensite plates.

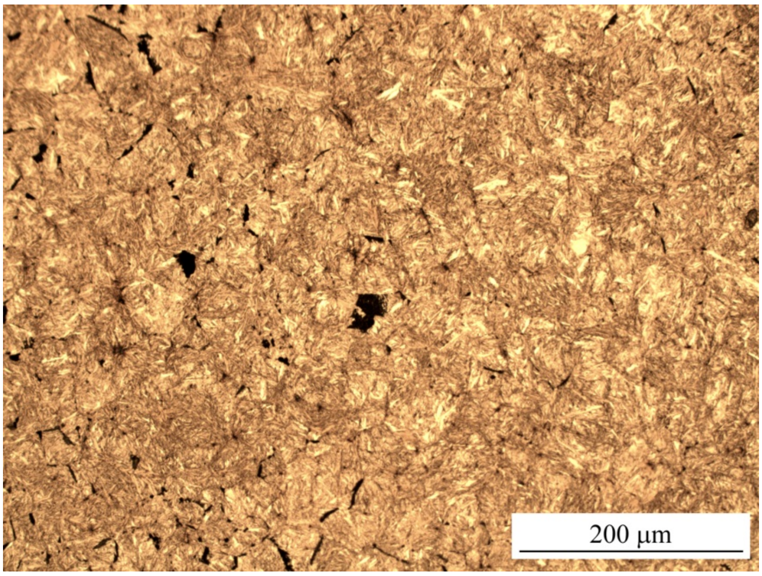

Figure 7 - Region of the diffusion couple in which the eutectoid decomposition ceases (~6.5%).

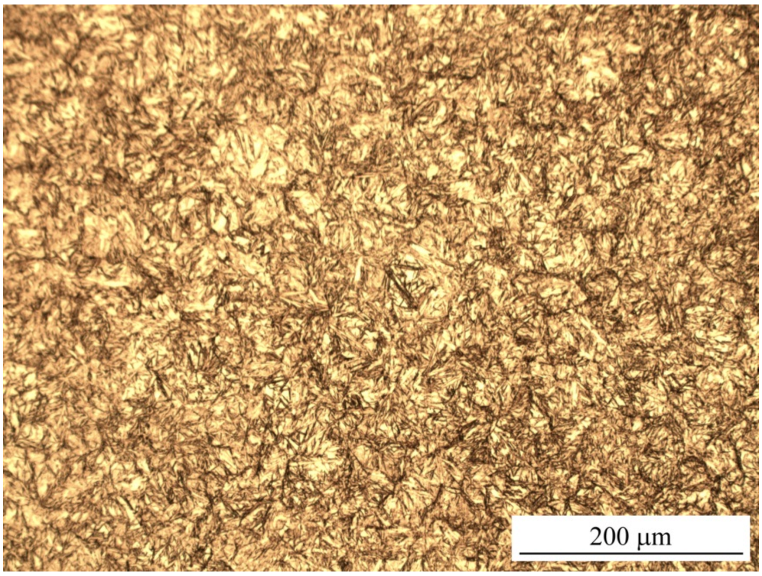

Figure 8 - High Ni region of the diffusion couple.

ISSN 1516-392X

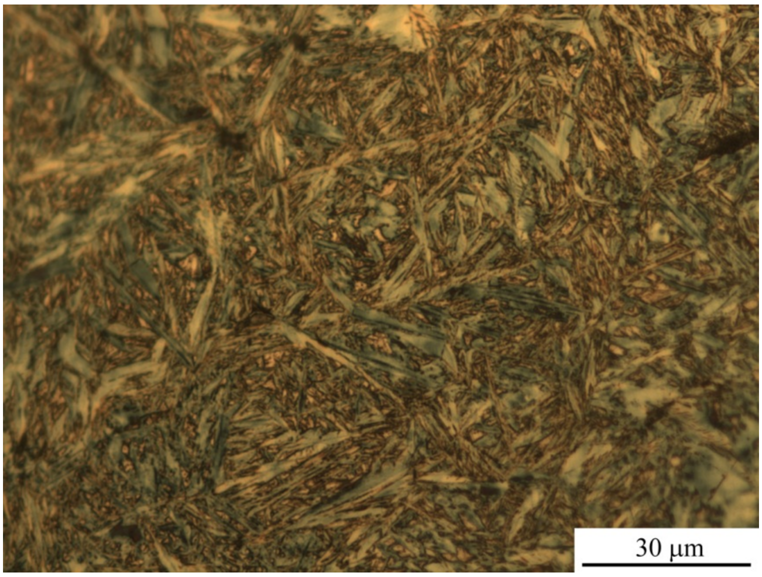

Figure 9 - High magnification view of 5%Ni region. The martensite plates are thin, and all the space between the larger plates is filled with smaller ones.

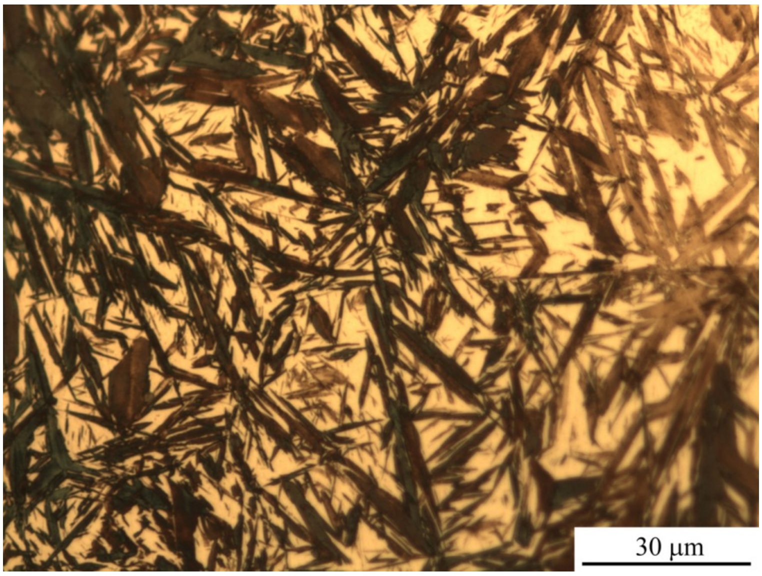

Figure 10 - High magnification view of high Ni region. The martensite plates are larger that those found at 5%Ni region, and there is a considerable amount of retained austenite.

4 CONCLUSIONS

As the nickel content rises, in oil quenched, 0.8%C samples, there is a significant change of scale in the martensite that is formed. Retained austenite dos not appear significantly until Ni contents of about 6.5%.
The use of diffusion couples provides a powerful tool to study transitions on behavior of phase transformation with the variation of alloy elements contents, as one single sample can provide a map of the material behavior as a function of the alloying element content.

REFERENCES

1 Kaufman, L. e Cohen, M. The Martensitic Transformation in the Iron-Nickel Sysytem. *Transactions of AIME.* 1956, Vol. 206, pp. 1393-1401.
2 Dunne, D. P. e Wayman, C. M. The crystallography of ferrous martensites. *Metallurgical Transactions.* 1971, Vol. 2, pp. 2327-2341.
3 Entwisle, A. R. The kinetics of martensite formation in steel. *Metallurgical Tranbsactions.* 1971, Vol. 2, pp. 2395-2407.
4 Hutchinson, C. R., et al., et al. A novel experimental approach to identifying kinetic transitions in solid state phase transformations. *Scripta Materialia.* 2004, Vol. 50, pp. 285-289.
5 Sinclair, C. W., Hutchinson, C. R. e Bréchet, Y. The Effect of Nb on the Recrystallization and Grain Growth of Ultra-High-Purity α-Fe: A Combinatorial Approach *Metallurgical and Materials Transactions.* 2007, Vol. 38A, pp. 821-830.