



# TRANSITIONS IN THE AUSTENITE DECOMPOSITION PRODUCTS IN Fe-5%Ni/Fe-10%Ni DIFFUSION COUPLES WITH 0.8%C<sup>1</sup>

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## 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. The sample used in this study was austenitized under vacuum in a quartz capsule at 900°C for 20 minutes and isothermally transformed at 400°C for 10 minutes in a Tin bath. The sample was finally quenched in still water at room temperature after isothermal treatment. In the lower nickel region, extensive formation of inverse bainite was observed, with some retained austenite and plate martensite filling sparsely distributed previously untransformed regions. As the Nickel content progresses, the amount of bainite formed decreases rapidly, and the high nickel region is completely untransformed during the isothermal treatment, and converted into plate martensite and retained austenite as the sample is rapidly cooled in water.

**Keywords:** Bainite; Austenite decomposition; Martensite; Morphological transitions.

## TRANSIÇÕES NOS PRODUTOS DE DECOMPOSIÇÃO DA AUSTENITA EM PARES DE DIFUSÃO FE-5%Ni/Fe-10%Ni COM 0,8%C

### Resumo

Um par de difusão composto de ligas Fe-5%Ni e Fe-10%Ni foi carbonetado em um banho de sal para o teor de 0,8%C ao longo da espessura. As amostras tinham um gradiente suave de Ni de 5 a 10%. A amostra usada neste estudo foi austenitizada a 900°C por 20 minutos em uma cápsula de quartzo selada sob vácuo, e posteriormente tratada isotermicamente a 400°C por 10 minutos em um banho de estanho fundido. A amostra foi então temperada em água após o tratamento isotérmico. Na região de baixo níquel, foi observada extensa formação de bainita inversa, com martensita maclada e austenita reitada preenchendo regiões esparsas em que a decomposição eutetóide não aconteceu. Com o aumento do teor de níquel, a quantidade de bainita formada cai rapidamente, e a região de alto níquel não sofre nenhuma transformação ao longo do tratamento isotérmico, sendo convertida em martensita maclada e austenita reitada quando do resfriamento rápido em água..

**Palavras-chave:** Bainita; Decomposição da austenita; Martensita; Transições morfológicas.

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## 1 INTRODUCTION

According to Aaronson et al.,<sup>(1)</sup> there are three definitions for bainite:

1) Generalized microstructural definition: According to that definition, bainite is a non-lamellar product of a non-competitive mechanism of eutectoid decomposition.

2) Overall reaction kinetics definition: bainite has an own C curve in the TTT diagram, below, but extensively overlapped with pearlite, yet entirely below the C curve of pearlite

3) Surface relief definition, according to which the ferrite plates have a surface relief effect associated to them, and grow by a martensitic mechanism or martensitic steps paced with carbon diffusion. This definition implies the need of two product phases.

As established by the microstructural definition, bainite and pearlite are two ends of a microstructural continuum; On the view that bainite is a non-lamellar product of eutectoid decomposition, the morphology of the ferrite should be considered irrelevant, and grain boundary allotriomorphs would also fall into that definition.

The idea that there is a symmetry in the decomposition of austenite around the eutectoid point has been around for a considerable amount of time,<sup>(2)</sup> but little attention to this concept has been given due to the enormous controversy over the mechanism of bainite formation when ferrite is the leading phase. The central point in this symmetry would be pearlite. When pearlite and bainite coexist, the scales of the ferrite differ by one order of magnitude.<sup>(3)</sup>

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.,<sup>(4)</sup> and has the great advantage of eliminating the need for production of several different alloys to study the influence of a certain alloying element.

## 2 EXPERIMENTAL PROCEDURES

Diffusion couples were produced from a Fe-5%Ni and a Fe-10%Ni alloys by hot cladding. The couples were solution treated at 1400°C for 36 hours to allow for the formation of the Ni gradient. The resulting Nickel gradient is about 0.2 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 carburized diffusion couple was austenitized at 900°C for 20 minutes and isothermally treated in a tin bath at 400°C for 10 minutes. The couple was finally quenched in still water at room temperature. The couples were encapsulated in quartz tubes under vacuum for all heat treatments except the carburization and isothermal hold at 400°C. At the end of the austenitization treatment, the quartz capsule was broken inside the furnace at 900°C for transfer of the sample to the tin bath. The sample was submitted to standard metallographic preparation procedures. The final polishing was done in 0.05 μm alumina suspension. Etching was done with a 3% nital solution. The samples were submitted to optical and electron microscopy analyses to evaluate the microstructure. Energy Dispersion X-Ray analyses (EDS) were conducted on selected points to determine the Ni content corresponding to transitions in behavior.

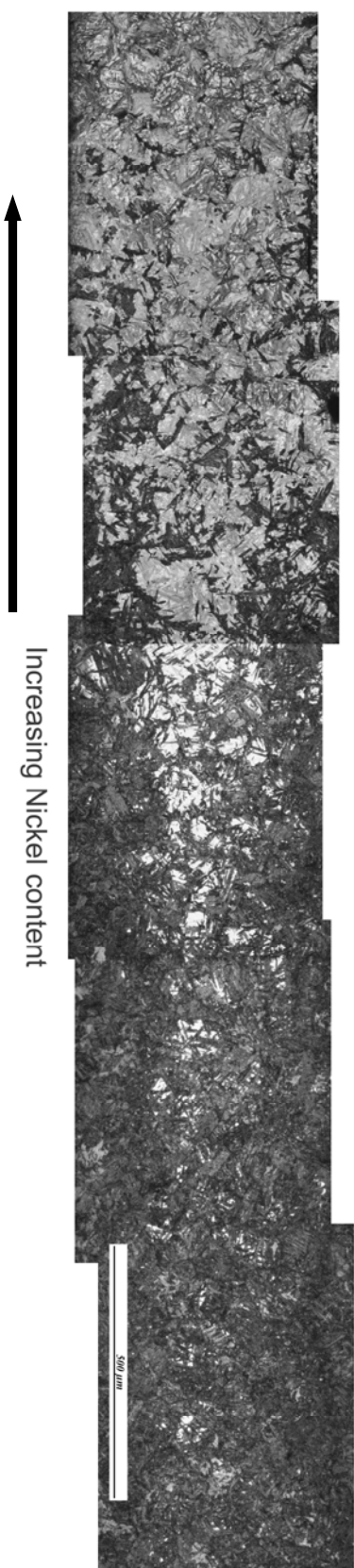
The chemical compositions of the two original alloys are given in Table 1.

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

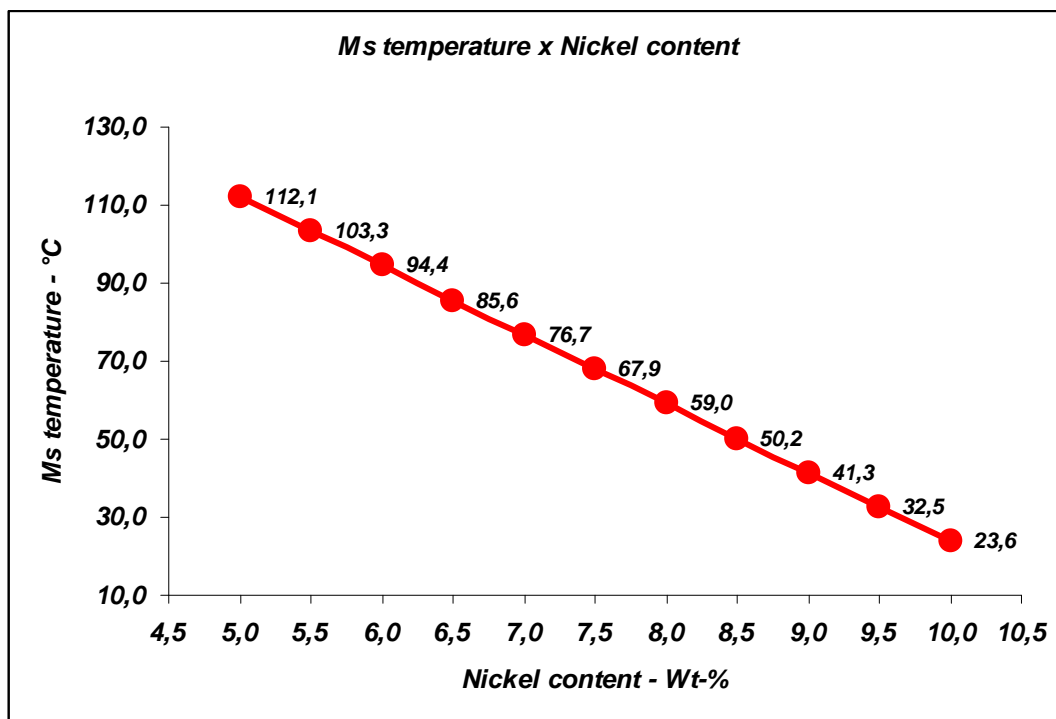
<b>Fe-Ni-5</b>	<b>C</b>	<b>Ni</b>	<b>Si</b>	<b>Mn</b>	<b>Cr</b>	<b>Mo</b>	<b>W</b>	<b>V</b>
	0,010	4,91	0,030	0,030	0,090	0,040	<0,010	<0,010
	<b>Ti</b>	<b>Nb</b>	<b>Co</b>	<b>Cu</b>	<b>Al</b>	<b>P</b>	<b>S</b>	<b>N<sub>2</sub></b>
	<0,0050	<0,010	0,010	0,050	<0,0050	<0,0050	0,001	0,0048
<b>Fe-Ni-10</b>	<b>C</b>	<b>Ni</b>	<b>Si</b>	<b>Mn</b>	<b>Cr</b>	<b>Mo</b>	<b>W</b>	<b>V</b>
	0,003	9,94	0,020	<0,010	0,090	0,040	0,030	<0,010
	<b>Ti</b>	<b>Nb</b>	<b>Co</b>	<b>Cu</b>	<b>Al</b>	<b>P</b>	<b>S</b>	<b>N<sub>2</sub></b>
	<0,0050	<0,010	0,010	0,050	<0,0500	<0,0050	0,001	0,0042

### 3 RESULTS AND DISCUSSION

An overview of the treated diffusion couple is given in Figure 1. The microstructure is composed of an eutectoid decomposition product, with non-decomposed regions in between. These regions were then transformed to martensite upon water quench, with some austenite being retained due to the low Martensite Start temperature ( $M_s$ ) of the high nickel region. This will be referred to as the M-A (martensite-austenite) constituent. The  $M_s$  along the diffusion couple was estimated using Andrew's formula, considering only the Nickel content. The calculated  $M_s$  is displayed as a function of Ni content in Figure 2. The martensite has a plate morphology, which is typical of high carbon-high nickel materials. In the higher nickel regions, butterfly martensite, as described by Umemoto and Tamura,<sup>(5)</sup> is largely observed. Butterfly martensite is shown in Figures 3 and 4.

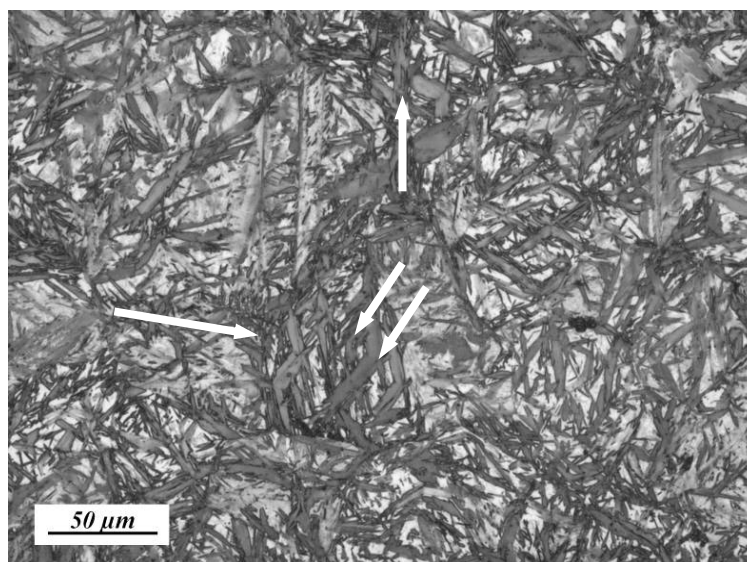


**Figure 1** - Overview of the diffusion couple after isothermal treatment and water quench.



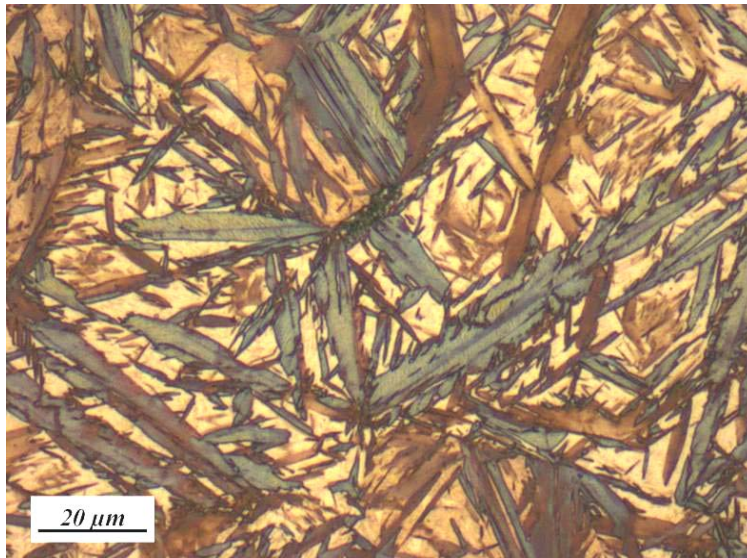
**Figure 2** - Ms temperature as a function of Nickel content along the diffusion couple, for a fixed carbon content of 0.8%.

As the Nickel content rises, the fraction of M-A constituent rises. The first significant delay of the eutectoid transformation takes place at approximately 6.8%Ni. The calculated Ms temperature for this region is about 80°C. The last island of eutectoid decomposition product takes place at a Nickel content of approximately 8.9%. At this region, the estimated Ms temperature is 43°C. From this point until the 10%Ni region, there is no readily detectable increase in the fraction of retained austenite.



**Figure 3** - Extensive formation of plate martensite in the 10%Ni region of the diffusion couple. White arrows point butterfly martensite.

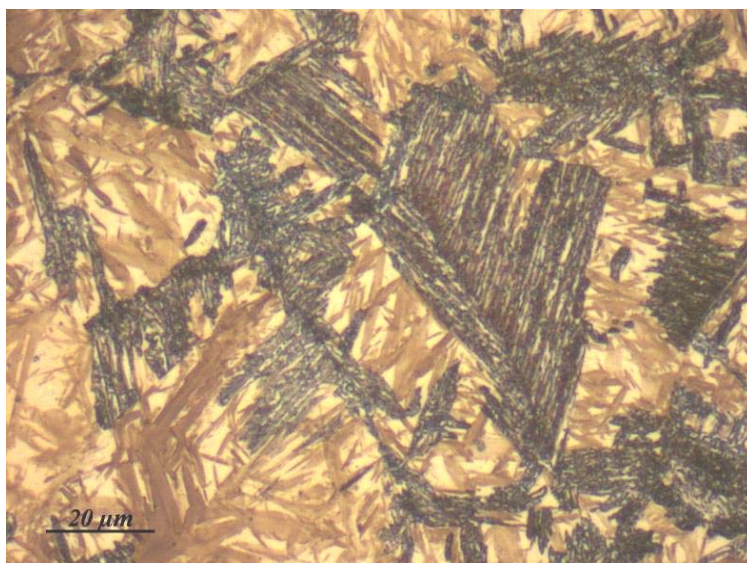




**Figure 4** - Higher magnification view of butterfly martensite.

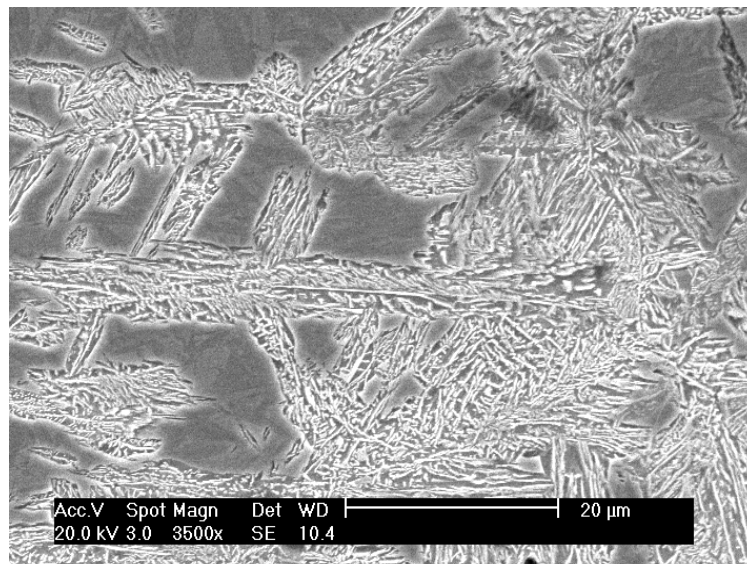
At high nickel contents, the  $M_s$  temperature is estimated as  $23.6^\circ\text{C}$ . Nevertheless, extensive formation of martensite is observed in the high nickel end of the diffusion couples. This inconsistency between calculated  $M_s$  and the real  $M_s$  can be explained by the fact that the  $M_s$  temperature is largely affected by the microstructure of the material prior to cooling and by experimental procedures. Grain size and carbide dissolution are known to influence the  $M_s$  temperature.<sup>(6)</sup> Also, this parameter is largely dependant on the experimental procedure. Different cooling rates may also affect the  $M_s$  temperature.

A high magnification view of a colony of eutectoid decomposition products is shown in Figure 5. The typical fernlike morphology that some authors attribute to bainite can be clearly seen in this figure. The angular distribution of eutectoid product can be an indication of two different phenomena: distinct nucleation events may have taken place on either side of the grain boundary, leading to eutectoid growth into both adjacent grains, or a single nucleation event originating a particle with dual orientation relationships may have occurred.



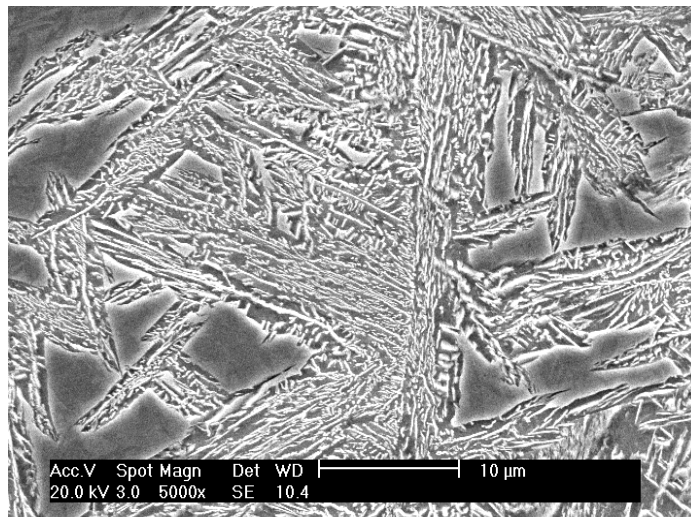
**Figure 5** - Eutectoid decomposition product towards the nickel rich end of the diffusion couple.

As to the nature of the eutectoid product, optical microscopy does not provide a reliable definition of what resulted from the decomposition of austenite. High magnification scanning electron micrographs of eutectoid product are shown in Figures 6 and 7. Extensive carbide formation is observed in the interior of the eutectoid product. The carbides are heavily aligned, forming recurring angles. This indicates that the eutectoid decomposition product is inverse bainite. A view of an eutectoid colony formed in a high nickel region (approximately 8.5%) is shown in Figure 8, and a higher magnification view of another colony formed in the same region is given in Figure 9. The distribution of carbides in these colonies indicates that this product is inverse bainite. The etching pattern surrounding the tip of the carbide indicates that cementite was the advancing phase at the moment that the reaction was halted by quench. The etching pattern largely resembles the schematic proposed by Kinsman and Aaronson<sup>(7)</sup> for the formation of inverse bainite. The formation of inverse bainite is in accordance with the principle of symmetry of the eutectoid decomposition of carbon steels recently rescued by Borgenstam et al.<sup>(2)</sup> The regions of the diffusion couple with a nickel content of approximately 8.0% are already mainly taken by M-A constituent, with sparse eutectoid colonies.

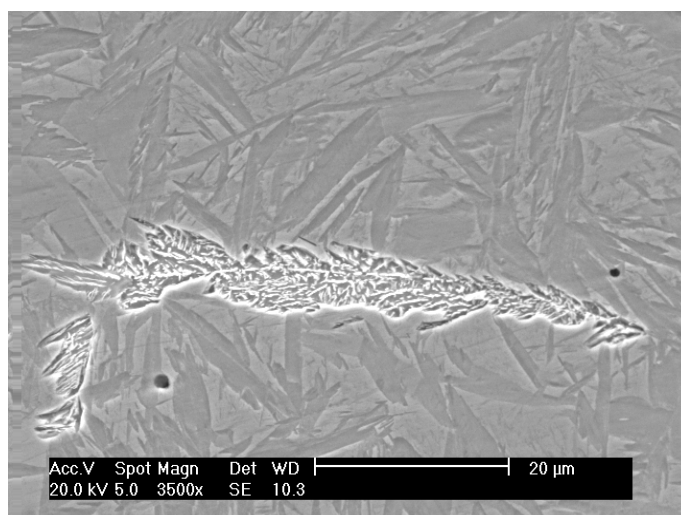


**Figure 6** - Scanning electron micrograph of an eutectoid colony towards the 5%Ni end of the diffusion couple; Extensive carbide formation is observed, with aligned carbide plates and recurring angles.

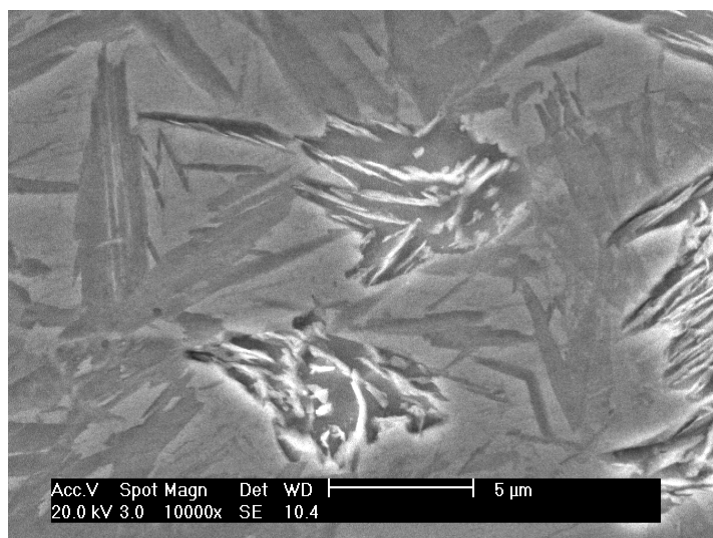




**Figure 7** - Higher magnification scanning electron micrograph of a eutectoid colony towards the 5%Ni end of the diffusion couple.



**Figure 8** - Eutectoid decomposition product near the 10%Ni end of the diffusion couple.



**Figure 9** - High magnification view of an eutectoid colony towards the 10%Ni end of the diffusion couple; The etching around the tip of the carbide plate indicates that cementite is the advancing phase.





### 3 CONCLUSIONS

The transitions observed along the diffusion couple are mostly kinetic in nature. The 5%Ni region of the couple is composed of a product of eutectoid decomposition and a mixture of plate martensite and retained austenite. The decrease in the amount of eutectoid product formation becomes more noticeable at a nickel content of approximately 6.8%, and eutectoid decomposition ceases completely at 9.0%Ni. The eutectoid product was seen to be inverse bainite. This observation is in accordance with the theory of symmetry of eutectoid decomposition recently rescued by Borgenstam et al.<sup>(2)</sup>

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