Металлофиз. новейшие технол. / Metallofiz. Noveishie Tekhnol.
2015, т. 37, № 4, сс. 531–537
Оттиски доступны непосредственно от издателя

Напечатано в Украине.

PACS numbers: 61.72.sd, 61.72.sh, 68.35.Dv, 81.20.Vj, 81.40.Ef, 81.70.Jb, 82.80.Ej

Influence of Heat Treatment on Spatial Distribution of Chemical Elements in Welding Microalloyed Steels

I. F. Tkachenko and M. A. Uniyat

Pryazovskyi State Technical University,
Department of Materials Science,
Universytets'ka Str., 7,
87500 Mariupol, Ukraine

The effect of isothermal holding of various duration at subcritical temperature on the spatial distribution of main chemical elements in the finally normalized X60, E36, and ASTM A516-65 steels is studied using SEM X-ray microanalysis. Based on results of statistical processing of the local concentrations' measurements, a quantitative characteristic for inhomogeneity of distribution is proposed. As revealed, the most uniform distribution of the elements is achieved after holding at (710±5)°C during 1–2 hours for X60 and ASTM A516-55 steels, and 4 hours for E36 steel. The obtained results are in agreement with the change in steels microstructure caused by the mentioned heat treatment.

Із застосуванням методи рентґеноспектральної аналізи в сканівному електронному мікроскопі вивчено вплив ізотермічної витримки різної тривалости за субкритичної температури на просторовий розподіл основних хемічних елементів у сталях X60, E36, ASTM A516-65 після остаточної нормалізації. На основі результатів статистичного оброблення одержаних даних рентґеноспектральної аналізи запропоновано кількісну характеристику неоднорідности просторового розподілу хемічних елементів. Встановлено, що найбільш однорідний розподіл хемічних елементів досягається після ізотермічної витримки при (710±5)°C тривалістю у 1–2 години для сталей X60, ASTM A516-65 і у 4 години для сталі E36. Одержані результати узгоджуються зі змінами мікроструктури досліджуваних сталей після аналогічних режимів термічного оброблення.

С применением метода рентгеноспектрального анализа в сканирующем электронном микроскопе изучено влияние изотермической выдержки различной продолжительности при субкритической температуре на пространственное распределение основных химических элементов в сталях X60, E36, ASTM A516-65 после окончательной нормализации. На основе результатов статистической обработки полученных данных рентгеноспектрального анализа предложена количественная характеристика неоднородности пространственного распределения химических элементов. Было установлено, что наиболее однородное распределение химических элементов достигается после изотермической выдержки при (710±5)°C в течение 1–2 часов для сталей X60, ASTM A516-55 и 4 часов для стали E36. Полученные результаты согласуются с изменениями микроструктуры исследуемых сталей после аналогичных режимов термической обработки.

Key words: spatial distribution of chemical elements, microalloyed steel, heat treatment, X-ray microanalysis.

(Received December 19, 2014; in final version, January 28, 2015)

1. INTRODUCTION

As of now, low-carbon microalloyed steels are the main type of materials for production of sheet and profiled steels for different purposes for application in civil engineering, shipbuilding, production of tubes for oil and gas pipelines [1]. Modern steels of this type are characterized by a high potential of operation properties, however, there are many problems, which arise during their production and cause corresponding types of embrittlement.

One of the most extended types of such embrittlement is connected with an inhomogeneous spatial distribution of structural components such as perlite colonies and particles of strengthening phases [2]. Well-known reason for formation of heterogeneities of microstructure is dendrite liquation, which causes formation of ferrite-perlite streakiness, which, in turn, induces anisotropy of mechanical properties, reduces strength level, decreases brittle fracture strength, causes premature localization of plastic flow, which results in decrease of the complex of operation parameters in the weld zone [3].

There is a number of production processes for thermic and thermomechanical treatments of steels, which make possible various types of the structural inhomogeneity [4]. At the same time, these technologies, including diffusion annealing, do not provide homogeneous distribution of chemical elements, particularly, because of interaction of their atoms with austenitic grain boundaries at high temperatures. In addition, methods of reliable quantitative estimation of the degree of homogeneity of the spatial distribution of chemical elements do not exist by now.

Obviously, formation of homogeneous distribution of chemical elements, especially Mn, V, C, Ti, Al, S, P, will promote essential increase and stabilization of rolled products quality due to homogeneous distribution of disperse structural component (colonies of perlite-like components) and particles of hardening phases (carbides, nitrides, and carbon nitrides) [5–9].

The goal of this work is development of the method for reliable quantitative characteristic of the degree of homogeneity of chemical elements distribution in microalloyed steels and investigation of the impact of thermal treatment on the developed parameter.

2. EXPERIMENTAL TECHNIQUE

Investigations are performed on the waste of specimens for mechanical testing, chosen from sheet products of X60 steel after controlled rolling, steels E36 and ASTM A516-65 in hot-rolled state. The specified treatments for each steel, which was performed under industrial conditions, are noted below as Treatment No. 1.

All investigation steels in the form of a card with dimensions of $200\times100\times18$ mm^3 were also subjected to the thermal treatment under semi-industrial conditions and investigation conditions. Complex thermic treatment, which includes isothermal holding at 710°C during one hour (Treatment No. 2), two hours (Treatment No. 3), and four hours (Treatment No. 4) with further normalization at optimal temperature of (710±5)°C for each investigated steel is used as such investigation conditions.

After thermic treatment under investigation conditions, specimens were cut out from the feedworks, and standard testing of their mechanical properties was carried out with further investigation of the degree of inhomogeneity of the chemical elements distribution. This investigation was performed using JSM-6490LV (Japan) scanning electron microscope with INCA Penta FETx3 (OXFORD Instruments) energy-dispersion attachment.

Local concentrations of the main chemical elements (C, Mn, Si, V, Gr, Ti, S, P, Mo, Ni, Al, Cu, Nb, Mg) in the investigated steels were de-

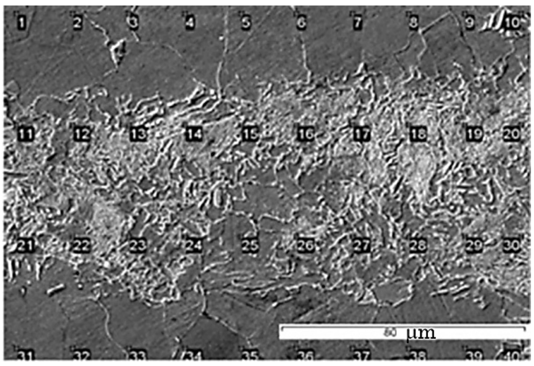

Fig. 1. Typical area of ASTM A516-55 steel microstructure in as-rolled condition with the points for measurement of local chemical elements concentrations.

termined in different points on the surface of a microslice (see Fig. 1). Investigated points were located in the form of rectangular network with a step between points, which exceeded typical dimension of the structural components.

3. RESULTS AND DISCUSSION

Results of the measurements of local concentrations of the chemical elements were treated according to the technique of two-factor dispersion analysis. Average dispersion by colons and lines were determined within the limits of the specified network, which intercross under different angles in order to exclude possible impact of orientation of colons and rows on the degree of inhomogeneity of concentrations distribution. After that, Fisher's criterion (F) for each chemical element was calculated as ratio of average dispersions of its concentrations in colons and rows. With consideration of corresponding degree of freedom, F_{cr} was found by Fisher–Snedecor distribution and obtained values of F and F_{cr} were compared under conditions of 95% confidence coefficient [10]. According to principles of dispersion analysis, ratio of $F/F_{cr} \geq 1$ attests to statistically significant difference of concentrations of chemical elements under investigation in different directions in space. It is proposed to use the value of $H = F/F_{cr}$ as quantitative parameter of the degree of inhomogeneity of spatial distribution of the chemical elements, which is statistically significant at $H > 1$ and $P < 5\%$.

Data for investigated steels concerning chemical elements, inhomogeneity of which substantially changes under influence of thermic treatment are shown in Figs. 2–4. For the rest of chemical elements such changes are not detected, that testifies to conservation of initial character of the spatial distribution under investigated conditions of their thermal treatment.

The impact of conditions of the thermal treatment on the degree of inhomogeneity of the chemical elements distribution in X60 steel is shown in Fig. 2. To begin with, it should be noted that substantial changes in character of spatial distributions of the following elements: C, Mn, S, Al, Mo, V, Ni, Cu under investigated conditions of the thermal treatment are observed. As we can see, in the initial state (Treatment No. 1) X60 steel is characterized by exclusively inhomogeneous distribution of all main chemical elements: $H > 1$. The distribution of such elements as S, Mo, Mn, C: H(S) = 1.8, H(Mo) = 1.7, H(Mn) = 1.6, H(C) = 1.5. Performance of the heat treatment under Treatment No. 2 resulted in substantial decrease of $H < 1$. The distribution of such elements as S, Mo, Mn, C: H(S) = 1.8, H(Mo) = 1.7, H(Mn) = 1.6, H(C) = = 1.5. Fulfilment of the thermal treatment under Treatment No. 2 condition resulted in substantial decrease of $H < 1$, in other words, in increase of homogeneity of the most part of the investigated elements.

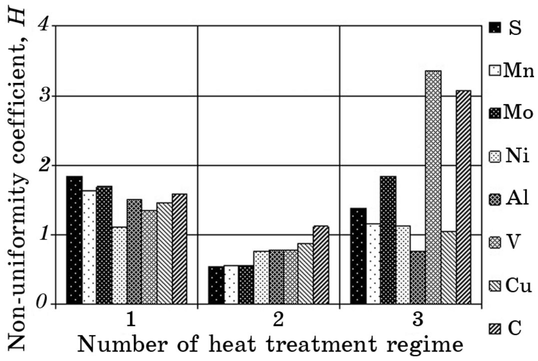

Fig. 2. Influence of the preliminary thermal processing on the non-uniformity coefficient H for the considered chemical elements in X60 steel.

From now on, after the heat treatment under Treatment No. 3 conditions, the substantial step-wise change of the inhomogeneity factor H is observed. Vanadium has the utmost inhomogeneous spatial distribution under the given condition: $H = 3.4$, and Al has the utmost homogeneous one: $H = 0.7$.

During the investigation of the impact of the thermal treatment condition on the character of the chemical elements concentration in the E36 steel, the corresponding substantial changes for the following elements were determined: C, Mn, S, Al, Ni, Cr (see Fig. 3).

As seen, in the initial hot-rolled state (Treatment No. 1), E36 steel has persistently high values of H factor for all investigated chemical elements, which points on their extremely inhomogeneous space distribution. Maximal H value is observed for C: $H(\mathrm{C}) = 14.8$, which corresponds to a high grade of ferrite-perlite stratification [6]. After the fulfilment of the investigation heat treatment under Treatment No. 2,

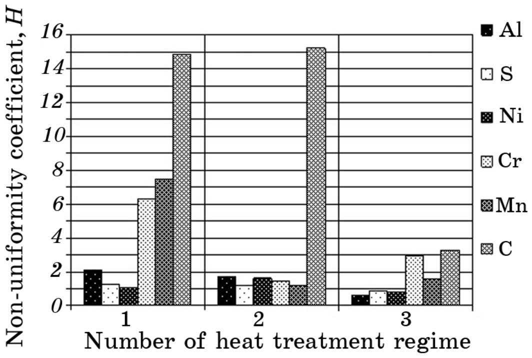

Fig. 3. Influence of the thermal processing modes on the non-uniformity coefficient H for the considered chemical elements in E36 steel.

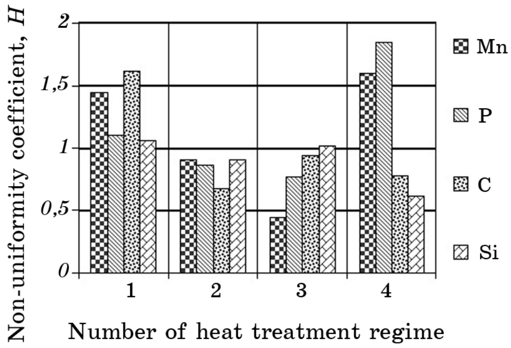

Fig. 4. Influence of the preliminary thermal processing on the non-uniformity coefficient H for the considered chemical elements in ASTM A516-55 steel.

changes were observed in the distribution of Mn concentrations distribution: value of H decreased from 7.4 to 1.2. The inhomogeneity factor H for the rest of the chemical elements conserved on the almost initial level. Results, obtained after the fulfilment of the heat treatment under Treatment No. 3 are close for the majority of the elements: $H \cong 1$. Based on the presented data, one can conclude that practically homogeneous concentration distribution of Al, S, and Ni after the heat treatment under Treatment No. 3 in the E36 steel takes place. At the same time, the completely homogeneous distribution is not achieved for Mn and C. Alongside with that, taking into account the observed tendency, one can expect the formation of the specified distribution at more long-termed holding.

For the ASTM A516-65 steel under the investigation conditions, changes in the character of the spatial distribution are observed for the following elements: Cu, Mn, P, and Si. As seen from Fig. 4, in the initial state (Treatment No. 1), the ASTM A516-65 steel is characterized by the inhomogeneous distribution of all investigated chemical elements: $H > 1$. The most inhomogeneous distribution takes place for C: H(C) = 1.6. Further increase of the holding duration to two hours does not result practically in the change of the achieved homogeneous distribution of the chemical elements. Increase of the holding duration until four hours (Treatment No. 4) leads to the dehomogenization of the chemical elements distribution of Mn and P and its conservation for C and Si. The data obtained verify the results of the metallographic investigations [5].

4. CONCLUSIONS

1. The quantitative characteristic of the degree of inhomogeneity of the space distribution of the chemical elements in steels, which is based

on the results of the statistical processing of the X-ray spectral measurements of the local concentrations in different directions on the surface of the microslice, is proposed.
2. The possibility of the formation of completely homogeneous distribution of the chemical elements in microalloyed welding steels after isothermal holding of various durations at subcritical temperatures is established.
3. The preservation of the achieved homogeneous character of the distribution during the final normalization of the steel takes place.
4. Obtained results are in agreement with the changes in the character of the microstructures of the investigated steels after heat treatment under similar operation modes.

REFERENCES

1. S. N. Golovanenko and N. M. Fonshtein, *Two-Phase Low Alloyed Steels* (Moscow: Metallurgiya: 1986) (in Russian).
2. E. Hornbogen, *Statistical Strength and the Steels Fracture Mechanics* (Moscow: Metallurgiya: 1986), p. 165 (Russian translation).
3. I. F. Tkachenko and K. I. Tkachenko, *Newsletter of the Priazov State Technical University: Collection of Research Papers*, Iss. 12: 90 (2002) (in Russian).
4. I. F. Tkachenko, *Newsletter of the Priazov State Technical University: Collection of Research Papers*, Iss. 10: 100 (2000) (in Russian).
5. I. F. Tkachenko and M. A. Uniyat, *Newsletter of the Priazov State Technical University: Collection of Research Papers*, Iss. 20: 105 (2010) (in Ukrainian).
6. I. F. Tkachenko and M. A. Uniyat, *Newsletter of the Priazov State Technical University: Collection of Research Papers*, Iss. 24: 86 (2012) (in Ukrainian).
7. *Method of the Heat Treatment of Metal Products from Structural Alloyed Steels*, Patent of Ukraine No. 65944 (МПК C 21 D 1/00) (in Ukrainian).
8. *Method of the Complex Heat Treatment of Metal Products from Alloyed Steels*, Patent of Ukraine No. 75610 (МПК C 21 D 1/00) (in Ukrainian).
9. *Method of the Heat Treatment of Metal Products from Normalizable Alloyed Steels*, Patent of Ukraine No. 83059 (МПК C 21 D 1/00) (in Ukrainian).
10. V. E. Gmurman, *Theory of Probabilities and Mathematical Statistics: Tutorial for HEI. 9-th Edition* (Moscow: Vysshaya Shkola: 2003) (in Russian).