Effect of phase shape and size on the FEM analysis of indentation test of duplex stainless steel microstructures

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Abstract

This study presents the effect of modelling the microstructure of duplex stainless steels with different geometric shapes and sizes on the indentation test results. In this study, an indentation test is modelled on duplex stainless steel (DSS) sheets. The actual and simulated microstructures are modelled with finite element program. Actual microstructure is modelled on SEM image of microstructure. In simulated microstructures, the austenite phase is modelled with four different geometric shapes: circle, small square, big square and triangle. While the indentation depth is found to be 25.80 μm experimentally, an average depth of 22.58 μm is reached in the simulations. Also, the effect of microstructures of different phase ratios on indentation behaviour is investigated. When the austenite ratio increases by 13%, it is observed that the indentation depth increases by 2.1% on average. In summary, the actual and simulated microstructures in different shapes and sizes give similar deformation results.

Keywords Microstructure modelling · Duplex stainless steel · FEM · Indentation test

1 Introduction

Stainless steels are high-alloyed steels with a minimum chromium content of 10.5 wt%. Historically, stainless steels have been classified by microstructure and are described as austenitic, martensitic, ferritic, or duplex (austenitic-ferritic). In addition, a fifth family, the precipitation-hardenable (PH) stainless steels, is based on the type of heat treatment used rather than the microstructure [1]. The stainless steel families have different mechanical properties. The martensitic stainless steels have high strength, but low ductility, whereas the austenitic stainless steels have lower strength but much higher ductility. The ductility of ferritic and duplex stainless steels (DSSs) lies between those of martensitic and austenitic alloys, with ferritic stainless steels having similar strength levels to the austenitic alloys. The strength levels of the DSSs are higher than those of the austenitic alloys, but lower than those of

the martensitic materials. DSSs have approximately 2 times higher yield strength than austenitic stainless steels due to their high nitrogen content in their chemical composition. Also, pitting and stress-corrosion cracking resistance of DSSs are equal or higher than austenitic stainless steel. On the other hand, DSSs have austenitic and ferritic phases at the same time in the same microstructure. Mostly the internal structures consist of 50% ferrite and 50% austenite. In summary, the DSSs carry good properties of both austenitic and ferritic stainless steels [2].

DSSs are subjected to a lot of metal forming work in recent years due to the dual-phase microstructure and superior mechanical properties. In literature, some studies were carried out on this subject. For instance, Yıldızlı [3], studied the impact toughness of welded DSS and austenitic stainless steel plates under cryogenic conditions. The results of this study indicated that the duplex–austenitic weldment has a greater impact toughness for the weld

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metal relative to the austenitic–austenitic weldment under cryogenic conditions. Fang et al. [4], investigated crack behaviour of the LDX 2101 DSS by the hot compression test. The researchers showed that microcracks initiated on the interface of austenite and ferrite and propagated within ferrite. Huang and Young [5], tested that the behaviour of cold-formed lean DSS square and rectangular hollow sections. Tensile coupon tests and stub column tests were done to obtain the metallic material properties of the test specimens. They revealed that the strength of DSS can be increased with work hardening. Schwarm et al. [6], investigated the deformation behaviour of the DSS by using nanoindentation and FEM model. They compared different analysis method to determine elastic properties by nanoindentation. They showed that the elastic modulus can be determined by FEM and that the experimental results are 6% different. Paro et al. [7], showed that DSS was a difficult material to process because of its high toughness, low thermal conductivity and high work hardening. Olden et al. [8], approached DSS modelling from a different perspective. They idealized the phase structure with a circular and ellipse like shapes. Three different phase shapes were used in their study and successfully simulated hydrogen diffusion in DSS. Tao et al. [9], investigated the local mechanical properties of ferrite and austenite in DSS. They carried out a series of nanoindentation tests under various loads. Also, they modelled ferrite and austenite phase to simulate nanoindentation test in ABAQUS. Their results show that the nanohardness of the ferrite phase (3.95 GPa) is lower than that of the austenite phase (4.1 GPa). After that study Tao et al. [10], studied about the modelling of hydrogen diffusion in DSS. The microstructure of DSS, consisting of austenite and ferrite phase, is successfully modelled in ABAQUS finite element program. Zhang et al. [11], were investigated mechanical properties of ferrite and austenite phases in DSS which is thermally aged under 475 °C. They used the nanoindentation test for mechanical properties and showed that the hardness of ferrite continuously increases with increasing aging time.

The finite element method (FEM) is a very cost-effective way to demonstrate the behaviour of a metallic material before it is used. Biwa and Storåkers [12], theoretically and numerically examined the indentation of a sphere. The researchers established a theoretical ground for early empirical formulas and showed the full-field analysis of the elastic plastic indentation. Mente and Boellinghaus [13], modelled the DSS microstructure to investigate the hydrogen-assisted cracking in the tensile test. They simulated the tensile test without microstructure and showed the stress distribution on the microstructure model with using tensile test simulation results. Their investigation revealed that the highest plastic deformation occurred

at austenitic phase. Ekici et al. [14], experimentally and numerically investigated the indentation behaviour of SiC particle reinforced metal matrix composites. In their study, random particle dispersion and size effect on the indentation depth and stress–strain distributions under the spherical indenter were studied. They specified that microstructure of metal matrix composites may be successfully modelled with FEM and the results showed good agreement with the experimental results. Ekici et al. [15], studied the effects of particle size and volume fraction on the indentation behaviour of particle reinforced metal matrix composites based on micro-structure models using the FEM. They specified that the indentation depth increased with increasing particle size and decreasing particle volume fraction. Wei et al. [16], investigated the size effects of microstructures in micro hydroforming. They used two different modelling techniques to model the microstructure, Voronoi model and real grain morphology model. The results revealed that the models may predict the local thinning in the critical area of the drawn cups due to size effects.

Indentation test is a common test method for estimation of mechanical properties of metallic materials. The main goal of indentation tests is to determine hardness and Young modulus of the materials. This test was used in several studies. Petryk et al. [17], investigated the estimation of power-law hardening exponent directly from indentation tests for anisotropic single crystal. They have modelled spherical indentation test and compared the results with the experimental data. The results of the study showed that the hardening exponent can be estimated with the experimental test of the spherical indentation. Calle et al. [18], determined the strain rate sensitivity of metallic materials by using a spherical indentation test at different velocities. They showed that the spherical indentation test can be used for mechanical characterization of metallic materials at high strain rates. Kannan et al. [19], investigated mechanical properties of inverse bainite with using diamond pyramid Vickers's indenter. They found that the Young's modulus and hardness of inverse bainite decreases and the ductility increases during the inverse bainitic transformation. Pham et al. [20], established a reverse algorithm for determining the plastic parameters of structural steel from the spherical indentation test. They proved that the plastic parameters can be determined by the spherical indentation test by comparing the results with the experiments. Wang et al. [21], tried to found plastic parameters of metallic materials such as hardening exponent and elastic modulus using spherical indentation. They used the indentation curve and obtain a good solution in the inverse analysis process. Also, they applied the approach to Al2024-t3 alloy and compared them with the uniaxial compression test data. Results showed that the new approach obtained good agreement with experimental

data. Wang et al. [22], estimated the elastic–plastic properties of metallic materials by using the indentation imprint. They measured the imprint and correlated the imprint with material parameters using mathematical analysis. According to results, especially references [8–10, 13–15], it was determined that the proposed methodology was effective and gave realistic results in engineering applications.

In this study, the microstructure of DSSs during the indentation test was modelled with finite element program, ABAQUS/Standard. The effects of the phase size and actual and simulated microstructures of DSSs on the spherical indentation test were investigated. Firstly, the stress–strain states and indentation depth were compared with actual and five different simulated microstructures simulation results. Then, simulated microstructures were created randomly distributed phase according to the austenitic and ferritic phase ratio.

2 Materials and numerical models

2.1 Duplex stainless steel

In this study, EN 1.4462 (UNS S32205) DSS was used for the indentation test. EN 1.4301 and EN 1.4016 were used modelling austenitic and ferritic phases, respectively. Their chemical compositions were analysed by an OBLF spark spectrometer. The chemical contents of the materials are given in Table 1. DSSs generally contain ferrite and austenite phases in equal proportions. Also, the microstructure of the DSS used in the study is shown in Fig. 1. For the metallographic examination, after a grinding and subsequent polishing process, the transverse microstructure was etched with a solution consisting of 30 ml HCl, 15 ml HNO₃, and 5 ml HF [23]. The microstructure image was taken from the scanning electron microscope (SEM). As seen from the image lighter grey phases are ferrite, darker grey phases are austenite.

SEM/EDS mapping was used for DSS to determine the composition of the austenite and ferrite phases (Fig. 2). General chemical composition of DSS was given in Fig. 2 with EDS images. On the other hand, chemical composition of austenite and ferrite phases were given in Table 2. Carbon content is ignored due to the contamination of vacuum chamber and sample. Chemical content of austenite and ferrite phases are similar. Chromium and molybdenum are slightly higher and nickel is slightly lower in the austenite phase than the ferrite phase.

Fig. 1 Microstructure of EN 1.4462

In order to perform a structural analysis with FEM program, the mechanical properties of the materials need to be known. In the ABAQUS program, the elasticity modulus of the material, true stress–true plastic strain values are needed for the structural analysis. For this reason, the tensile test was performed on the DSS and the relevant parameters were determined. Mechanical properties of the DSS were determined using ASTM-E8/E8M-15a tensile test standard [24]. Tensile tests were performed by an INSTRON tensile tester, which has a maximum 100 kN loading capacity, with monotonic loading at a cross head speed of 10 mm per min. The strain values were determined with a mechanical extensometer. Although the mechanical properties of DSS can be determined by tensile testing, the mechanical properties of austenitic and ferritic phases cannot be determined by tensile testing. Therefore, not only the DSS was tested by tensile test but also austenitic and ferritic stainless steels were tested for the modelling of ferrite and austenite phases. EN 1.4301 (AISI 304) and EN 1.4016 (AISI 430) stainless steel grades were used for tensile tests of austenitic and ferritic stainless steel, respectively. In the tensile test result, engineering strain and engineering stress curves were obtained. These curves were converted to true strain and true stress curves using Eqs. (1) and (2):

$$\varepsilon_t = \ln(\varepsilon_{eng} + 1) \text{ mm/mm} \quad (1)$$

$$\sigma_t = \sigma_{eng} \cdot (\varepsilon_{eng} + 1) \text{ MPa} \quad (2)$$

where ε_t is the total true strain, ε_{eng} is the engineering strain, σ_t is the true stress, σ_{eng} is the engineering stress.

Table 1 Chemical composition (weight %) of stainless steels

Elements	C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	Ti	Ce
EN 1.4462	0.016	0.41	1.34	0.022	0.001	22.22	5.71	3.12	0.169	0.21	0.005	0.015
EN 1.4301	0.08	0.72	1.69	0.043	0.008	18.10	7.91	0.30	0.112	0.43	0.018	0.002
EN 1.4016	0.12	1.00	1.00	0.04	0.03	16.54	0.30	0.02	0.01	0.12	0.009	0.001

Fig. 2 Scanning electron microscope image and EDS mapping of EN 1.4462

Table 2 Chemical composition (weight %) of austenite and ferrite phases of stainless steels

Phases	Si	V	Cr	Mn	Ni	Mo	Fe
Austenite (Spectrum 3)	0.56	0.24	23.87	1.47	4.95	3.47	65.45
Ferrite (Spectrum 2)	0.58	0.00	22.02	1.48	6.70	2.71	66.51

The true stress and true strain curves obtained these equations are given in Fig. 3 and also general mechanical properties are given in Table 3. The plastic strain values are also obtained by subtracting the elastic strain from the total true strain values, Eq. (3):

$$\epsilon_{pl} = \epsilon_t - \epsilon_{el} = \epsilon_t - \frac{\sigma_t}{E} \quad (3)$$

where ϵ_{pl} is the plastic strain, ϵ_{el} is the elastic strain which is calculated from Hooke Law and E is the Young's (elastic) modulus. Also, Holloman equation was used to show non-linear effect in model, as seen in Eq. (4).

$$\sigma = K \cdot \epsilon^n \quad (4)$$

Fig. 3 True stress–true strain curves of the stainless steels **Table 3** Materials mechanical properties

Properties	EN 1.4462	EN 1.4301	EN 1.4016
Young's modulus, E (GPa)	202.6	210.3	192.1
Poisson's ratio, ν	0.3	0.29	0.32
Yield strength, $R_{p0.2}$ (MPa)	680	282	279
Tensile strength, R_m (MPa)	852	651	464
Strain hardening exponent, n	0.090	0.290	0.152
Strength coeff., K (MPa)	1155	1230	760

In this equation, σ is the true stress, K is the strength coefficient, ϵ is the true strain, n is the strain hardening coefficient.

2.2 Numerical model

Today, FEMs are often used to save time, reduce time-consuming and costly trial-and-error methods. In the models used in this study, it is aimed to determine the closest result as soon as possible and with the least effort. For this reason, 5 different microstructure models were compared with each other. All numerical simulations were done in the ABAQUS/Standard program [25]. ABAQUS permits parametric modelling and works well with other CAD programs. Besides, by writing subroutines within the program, the user can make the necessary arrangements.

Fig. 4 Black and white image of the microstructure

3D microstructure modelling is more realistic and convenient to understand material behaviour, but requires a high amount of CPU capacity in the virtual environment. Because the models made have high number of elements and nodes, which significantly increases the solution time. The study is simplified and modelled on 2D SEM image $0.6 \times 0.3 \text{ mm}^2$ part of the prepared specimen which can be clearly selected phases are displayed and taken the photograph under SEM, as seen in Fig. 1. The SEM image was first converted to black and white using an image processing program (Fig. 4). Black and white image was vectorised by the same image processing program. This

vectorization allowed to transfer of microstructure to the ABAQUS. In ABAQUS, the microstructure sketch was edited to close gap and deleted one of overlapping lines in same region. The partition of the face does not occur in these regions when the sketch has gaps or overlapping lines.

The non-linear FEM was used in the indentation test and microstructure modelling. This method considers the non-linearity that results from geometric, material and indenter contact with the surface. The duplex microstructure was modelled using a 3-node axisymmetric element type (CAX3) (Fig. 5a). Thus, the model had 22,410 elements and 11,416 nodes. The indenter was meshed using 2-node axisymmetric rigid (RAX2) elements. The indenter is allowed to move only in the Y-axis direction. Only half

of the microstructure model is modelled since the indentation problem is axisymmetric. Therefore, axisymmetric boundary conditions are applied to all nodes along the Y axis. The bottom edge of the microstructure model is fixed.

In Fig. 4, the white parts are modelled as ferritic, while the black parts are modelled austenitic. The phases in the modelling are assumed to sliding against each other and the damage that occurs in phases is not taken into consideration. The indenter which has 0.794 mm diameter was contacted to the upper surface of the microstructure. The contact type was determined from surface to surface and the friction coefficient was selected as 0.3. Indentation analyses were repeated under the same conditions by changing the phase shapes. In the repeated analyses,

Fig. 5 Actual and simulated microstructures for specimens with different phase geometry. **a** Actual microstructure, **b** Circle based microstructure ($D=10\text{--}20\text{ }\mu\text{m}$), **c** Square based microstructure

($L=20\text{ }\mu\text{m}$), **d** Big square based microstructure ($L=50\text{ }\mu\text{m}$), **e** Triangular based microstructure ($L=50\text{ }\mu\text{m}$)

phases in the microstructure are created by using a code developed by the authors for known geometric shapes. The geometric shapes used are square with a 20 μm edge length (Fig. 5c), circles with a diameter between 10 and 20 μm (Fig. 5b), non-contact squares with a 50 μm edge length (Fig. 5d) and triangular with a 50 μm edge length (Fig. 5e). The geometric shapes are randomly distributed in the sample borders. In models made with small squares and circles, shapes are allowed to intersect each other. These intersections caused larger areas than non-contact squares or circles. The lines that are located in the intersected areas are trimmed. All analyses were performed using a Rockwell T-scale hardness test with a 150 N compression load on indenter. As the test results, penetration depth was used instead of hardness value. Thus, the simulation results are compared directly with the experimental results.

Mesh sizes were chosen 0.005 mm for all FE models. Also, smaller sizes than the chosen mesh sizes were tried and it showed that the smaller mesh sizes did not change the results but increased solution time. On the other hand, bigger mesh sizes gave a different result which is not proper.

3 Results and discussions

In this study, different analyses were made for 5 different microstructure models using small squares, large squares, circles, triangular and actual microstructure. The actual microstructure image was analysed in an image processing program to determine the austenite and ferrite ratios which are 62% and 38% for selected region, respectively. This ratio was taken into consideration in all microstructure models created. In all models, the indenter was removed from the surface after loading. The indentation depths obtained experimentally and numerically were given in Table 4. The error rates of the analyses result according to the experimental results were also given in the same table. The most important factor affected the indentation

results were only the tensile test results which were used in material modelling. Therefore, in the microstructure models, both the ferritic and the austenitic stainless-steel tensile test results were used while material modelling in the program.

The average indentation depth of these analyses was 22.58 μm . When the average value was considered, it was generally seen that the standard deviation between microstructure models was 0.48 μm (2%). Therefore, it was observed that the results of the analysis made with the different phase shapes were similar. On the other hand, the similarity of the analysis results suggested that microstructure models with simpler geometries could be used instead of actual microstructure models. Modelling and meshing of actual microstructure models in a computer environment are very complex and time consuming. Instead of actual microstructure modelling, modelling and solution time can be decreased with using microstructure models with simple geometric shapes.

The effect of the phase ratio in the microstructure model on the indentation depth was studied. Random phase ratios were created in microstructure models consisting of small squares and large squares and the indentation depths were compared. These values were given in Table 5. In the analysis, it was seen that the indentation depth increases with the increasing austenite phase in the microstructure. The fraction of austenite phase, which is a softer phase compared to the ferrite phase, in the microstructure affects the overall hardness of the model as expected. Figure 6 clearly showed that even though the austenite fraction in the microstructure was the same, higher indentation depth was obtained in the microstructure models using larger squares. For the austenite phase fraction of 46.7%, 56% and 61%, the depth of change in different microstructure models were 1.8%, 0.4% and 1.3% respectively. These rates of change show that there was no significant difference between the phase shapes in the microstructure in the finite element models. The most basic parameters affecting the indentation depth are the phase ratios in the model.

Table 4 Experimental and numerical indentation depths (μm) and phase ratio

Model Type	Ferrite–Austenite (%)	Indentation depth (μm)	Error (%)
Actual	38.5–61.5	22.19	14.0
Triangular phase	39.9–60.1	22.51	12.8
Circular phase	39.7–60.3	22.64	12.2
Square phase	39.0–61.0	22.65	12.2
Big square phase	39.0–61.0	22.95	11.0
Experimental	38.5–61.5	25.80	–

Table 5 Variation of indentation depth depending on microstructure model type and austenite ratio (μm)

Model type	Austenite fraction (%)	Indentation depth (μm)
Square phase	47.4	22.11
	56.7	22.48
	61.0	22.65
Big square phase	47.4	22.53
	56.7	22.58
	61.0	22.95

Fig. 6 Effect of phase type and austenite fraction (A_f) on indentation depths (μm) based on the simulation model

The equivalent residual stress (von Mises) distribution on the sample after the indentation test was given in Fig. 7. The stress distribution is generally concentrated around the indenter and below the indenter in all of the models. In the actual microstructure model given in Fig. 7a, the equivalent stress appeared to be concentrated around the indenter. Equivalent stress was higher in the ferritic phase, which showed higher resistance to deformation. Higher stresses were formed under the indenter as well as around the indenter (Fig. 7b–e). The maximum stresses were concentrated in the ferritic region. The austenitic phase deformations were prevented by the ferritic phase and thus the stress on the ferritic phase increases. However, as in other models, the maximum stresses were concentrated around and below the indenter. It was observed that as the distance between the austenite phases increases, or in other words, as the ferrite ratio increases, the stress density increases under the indenter. Generally, in all models, high stress occurred in the mesh is between the indenter surface and the sample surface. These stresses depend on the contact type and friction coefficient. In the analysis, it was difficult to converge the program solution, especially in the first iterations of contact. For this reason, contact control is applied to all models so that first iterations can be converged more easily.

In Fig. 8a, indentation profiles of different microstructure models were given with experimental result. The trend of indentation profiles was generally similar. In models which are modelled different geometric shapes, the profiles were very similar and appear to overlap. The accumulation of material at the junction of the

indenter and the upper surface of the sample was seen. This accumulation of material decreases linearly as you move away from the indenter, and passes through some negative area at the end of the sample. In Fig. 8b, microstructural models modelled with large squares and small squares were given the indentation profiles according to different austenite ratios. It can be said that the austenite fraction and geometric shape are not generally affected in the indentation profiles. However, it can be said that the increase in the austenite fraction and the phase size, increases the indentation depth a little. Additionally, although the softer austenite phase is easily deformed while the indenter is advanced, but the ferritic phase which is relatively harder, reduces this deformation.

4 Conclusions

In this study, the microstructure of DSS is modelled with finite elements using different geometric shapes for phases, and the behaviour of the indentation test is investigated in two dimensions. The sample microstructure is imaged by SEM and the actual microstructure image is modelled by the finite element program. The effect of different phase geometries, the phase ratio and phase size on the indentation depth and stress distribution is investigated.

The indentation depth and the stresses were compared with each other as a result of the analysis. As a result of the study, the indentation depth values were in good agreement with the experimental results. The closest result was obtained from the large square phase model with 11% error compared to the experimental one. The results of the simple geometric modelling and actual microstructure model are similar and only has 1% difference for the indentation depth. For this reason, it has been determined that, instead of the actual microstructure model, finite element models with appropriate phase ratio modelled with simpler geometric shapes can be used. Thus, it has been shown that it is easy to get realistic results with a model that is created more easily and quickly.

It was observed that the austenite phase, which was soft, was not easily deformed because of the surrounding ferrite phase. Therefore, it was determined that the stress accumulation was on the ferrite phase. In future work, this modelling technique can be used to model microstructure behaviour in the tear zone during DSS forming. Literature can be made about the tearing damage by examining the crack initiation and progress under forming processes.

Fig. 7 Equivalent residual stress (von Mises) after indentation test based on numerical microstructure model: **a** Actual microstructure, **b** circle based microstructure, **c** square based microstructure, **d** big square based microstructure, **e** triangular based microstructure

Fig. 8 **a** The effect of different microstructure model on the indentation profiles, **b** the effect of different austenite fraction on the indentation profiles

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Compliance with ethical standard

Conflict of interest The authors declare that they have no conflict of interest.

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