Formation Mechanism of Nanoscale Al_3Fe Phase in Al-Fe Alloy During Semisolid Forming Process

XIANG WANG, REN-GUO GUAN, and YU WANG

The formation mechanism of nanoscale Al_3Fe phase in Al-1Fe (wt pct) alloy during rheo-extrusion was investigated, and the mechanical property of the prepared alloy was also measured. The results show that the average length of Al_3Fe phase in Al-1Fe alloy prepared by rheo-extrusion is 300 nm, which is much more refined than the needlelike Al_3Fe phase in as-cast Al-1Fe alloy (50 μm). In rheo-extrusion, Al_3Fe phase formed by eutectic reaction is bonelike, but it could be continuously refined by the shear deformation in the wheel groove, in equal channel angular flow, and in expansion extrusion mold. The total equivalent strain of the shear deformation is higher than 4.82. The tensile strength and elongation of Al-1Fe alloy prepared by rheo-extrusion are 135 MPa and 30 pct, respectively. The tensile strength of Al-1Fe alloy prepared by rheo-extrusion is 58.8 pct higher than that of as-cast Al-1Fe alloy, and the elongation is 19 pct higher than that of as-cast Al-1Fe alloy. Compared with as-cast Al-1Fe alloy, the improvements of tensile strength and elongation caused by shear deformation in rheo-extrusion are higher than the reported improvements induced by rare earth modification.

https://doi.org/10.1007/s11663-018-1323-7

I. INTRODUCTION

ALUMINUM alloys are widely used in automotive, aerospace, and transportation because of their low density, high specific strength, and excellent corrosion resistance.[1–4] However, iron in Al alloys always results in the formation of Fe-bearing phase, and the shape of Fe-bearing phase is generally needlelike. The needlelike Al-Fe phase easily causes stress concentration and dissevers Al matrix during plastic deformation.[5,6] Further, Fe is one of the most common elements in Al alloys, and the solid solubility of Fe in Al is relatively low.[7,8] In order to avoid the formation of Fe-bearing phase, the most ideal method is to reduce the content of Fe in Al melt to an extremely low level, or even to completely remove Fe from Al melt. However, the fact is that Al melt is often exposed to Fe container during the forming process, and the inclusion of Fe in Al is almost inevitable; besides, the Fe removal process is complex and costly. Also, it has been reported that the needlelike Fe-bearing phase forms in Al alloys even if the content of Fe is only 0.1 wt pct,[9] but the content of Fe in some recycled Al alloys can be as high as 2 wt pct. Therefore, Fe has become a key factor in restricting the performance of Al alloys and limiting their application.

In recent years, the research topic has shifted from removing Fe to refining the Fe-bearing phase. It has been widely reported that Fe-bearing phase can be effectively refined and its morphology can be changed by some measures, and the negative effect of Fe-bearing phase can be partly avoided and Fe can even serve as a reinforcing particle.[9] The measures can be divided into two categories. One is refining the Fe-bearing phase during liquid forming and includes magnetic stirring,[10,11] rapid solidification,[12–14] element modification,[9] and so on. Ban *et al*. studied Al_3Fe phase in hypereutectic Al-Fe alloy during solidification under the function of magnetic field and found that Al_3Fe phase could be refined by AC magnetic field, while DC magnetic field had the opposite effect.[11] Li *et al*. found that primary Al_3Fe phase was refined because the nuclei density of primary Al_3Fe was increased by the magnetic field.[15] However, the magnetic field that can effectively refine Al_3Fe phase has a high requirement of magnetic field strength and stability, so it is costly and not suitable for large-scale industrial application. Some studies showed that Al_3Fe phase could be refined by rapid solidification because the solid solubility of Fe in Al matrix was expanded by increasing the cooling rate.[16] Nevertheless, the research of Zhang *et al*.[17] indicated that Al_3Fe phase in Al-Fe alloy was more nucleation dependent than growth dependent (theory of

XIANG WANG and YU WANG are with the School of Materials Science and Engineering, Northeastern University, Shenyang 110819, China. REN-GUO GUAN is with the School of Materials Science and Engineering, Northeastern University and also with the School of Materials Science and Engineering, Northwestern Polytechnical University, Xi'an 710072, China. Contact email: 15840319082@126.com
Manuscript submitted August 5, 2017.
Article published online July 10, 2018.

Meredith). In fact, metal prepared by rapid solidification is generally in small scale and the process is of high cost. Several reports showed that the preferential growth of Al_3Fe phase could be retarded by the addition of an extra element, such as rare earth (RE), and Al_3Fe phase could be refined. However, the refinement effect of Al_3Fe phase induced by the added element is not obvious, and the addition of an extra element may introduce undesirable second phase and raise the cost. However, though Al-Fe phase was refined during liquid forming, both the tensile strength and elongation of Al alloys were not significantly improved. In the case of RE modification, the tensile strength of as-cast Al-1Fe (wt pct) alloy only increased from 85 to 89 MPa, and the elongation increased from 25.5 to 28.5 pct.[9]

Another measure is refining Fe-bearing phase by the shear stress in plastic deformation, especially multipass severe plastic deformation (SPD).[18] Repetitive continuous extrusion,[19] equal channel angular pressing (ECAP),[20] and high-pressure torsion[7,8] have been proved to be able to refine Fe-bearing phase. However, multipass SPD limits the production efficiency and the product is always in small scale. Though many methods have been proposed to refine Fe-bearing phase in Al alloys, some of them are not suitable for large-scale industrial application, while the others require strict processing parameters and are of high cost. No desirable measure that is cost-effective and applicable for industrial production to refine Fe-bearing phase has been put forward until now to the best of our understanding.

Semisolid forming is a novel metal processing technology, which is different from liquid forming and solid plastic deformation. Semisolid slurry with high solid fraction exhibits good fluidity and viscosity, and shear stress induced by slurry flow may achieve the refinement of brittle primary Fe-bearing phase in semisolid slurry. However, there have been few reports about the refinement of Fe-bearing phase in semisolid forming process so far. Rheo-extrusion is a typically semisolid forming method, and it can be used to prepare profiles with different sections of Al alloys, Mg alloys, *etc*. Recently, a new SPD method called "accumulative continuous extrusion forming (ACEF)" was developed based on rheo-extrusion, and it has been reported that α-Al grains in Al-Sc-Zr alloy, Al-Er alloy, and Al-Si alloy could be obviously refined by ACEF.[21–23] So, it can be deduced that the brittle Fe-bearing phase may be effectively refined by the shear deformation in rheo-extrusion, and the main purpose of this article is to investigate the refinement of Fe-bearing phase in the semisolid forming process and to provide new direction for how to handle the Fe in Al alloys.

II. EXPERIMENTAL PROCEDURES

Pure Al ingot (99.99 wt pct) and Al-10Fe (wt pct) master alloy were used to prepare the Al-1Fe alloy studied here. First, pure Al ingot was melted in a crucible resistance furnace under the protection of argon gas and then Al-10Fe (wt pct) alloy was added into the melt when the pure Al was heated to 730 °C and held for 10 minutes. A preheated ceramic rod was used to stir the melt in order to homogenize Fe. After degassing and deslagging, the melt was poured into the roll groove of a DZJ350 continuous rheo-extrusion machine to prepare Al-1Fe alloy rod with a diameter of 10 mm, and the schematic of rheo-extrusion is shown in Figure 1.

Samples for optical microscope (OM) and scanning electron microscope (SEM) observation were polished and etched by Keller reagent (2.5 mL HNO_3 + 1.5 mL HCl + 1.0 mL HF + 95 mL H_2O). An Olympus DSX500 metallographic microscope was used to observe the microstructure. A field emission–scanning electron microscope (Ultra Plus, Carl Zeiss, Germany) equipped with an energy-dispersive X-ray spectroscopy (EDS) instrument was used for microstructural observation and chemical component analysis. Specimens were investigated using an X-ray diffractometer (X' Pert Pro (Philips, Netherlands), Cu K_α irradiation, and λ = 0.15406 nm) to identify the second phase. Plate with ~ 500-μm thickness was cut from the prepared rod by electrospark wire-electrode cutting for transmission electron microscopy (TEM) specimens, and then it was mechanically polished from both sides to a final thickness of ~ 70 μm. Subsequently, foils with a diameter of 3 mm were punched and twin jet electropolished in a solution of 2.5 vol pct $HClO_4$ in ethanol at − 45 °C and 40 V. The TEM observations were performed using a TEM instrument (Tecnai G2 20, FEI) operated at an accelerating voltage of 200 kV. A CMT5105 electronic tensile testing machine was used to test the mechanical property at room temperature, the crosshead speed was set as 2 mm/min, and three samples were tested for each condition.

III. RESULTS

A. *Al-Fe Phase in Al-1Fe (Weight Percent) Alloy Prepared by Rheo-Extrusion*

The TEM graph of Al-1Fe alloy prepared by rheo-extrusion is shown in Figure 2(a). It can be found that massive second phase forms in Al-1Fe alloy during rheo-extrusion forming, and its average length is 300 nm. The nanoscale phase is composed of Al and Fe according to the EDS analysis shown in Figure 2(b) of

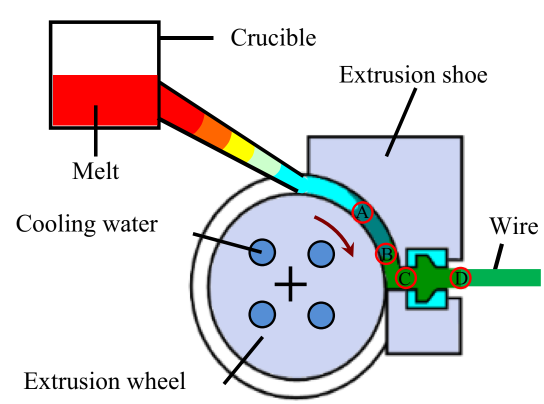

Fig. 1—Schematic of rheo-extrusion and location of sample taking.

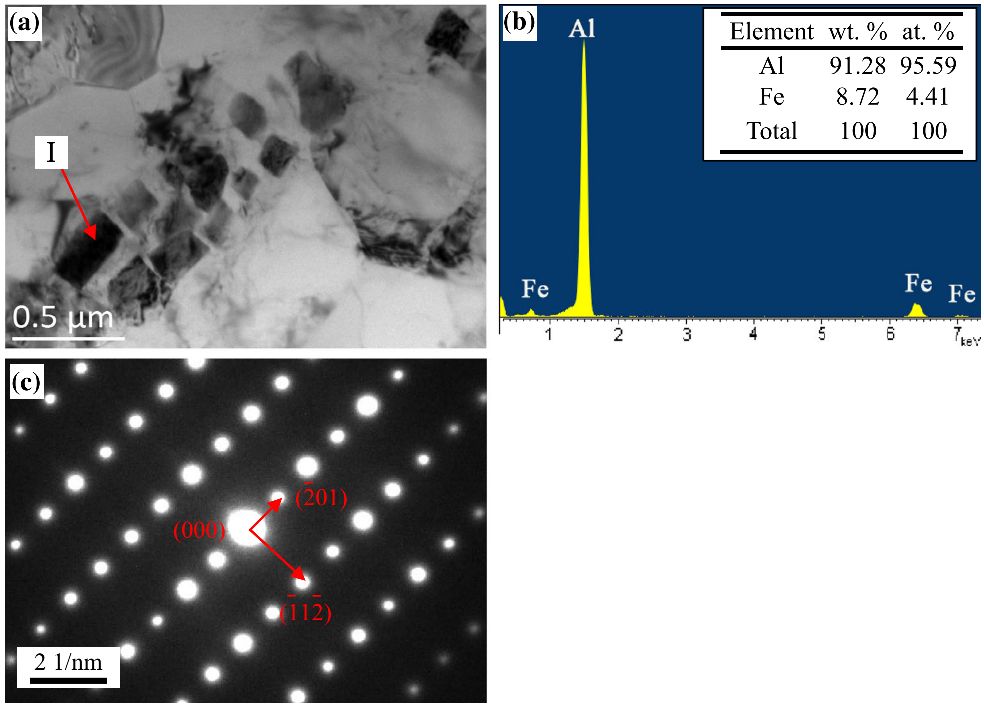

Fig. 2—(*a*) TEM graph of Al-1Fe (wt pct) alloy prepared by rheo-extrusion and (*b*) EDS analysis of point I in (a). (*c*) Corresponding SAED of point I, showing the bean direction was [1 5 2] of Al_3Fe phase.

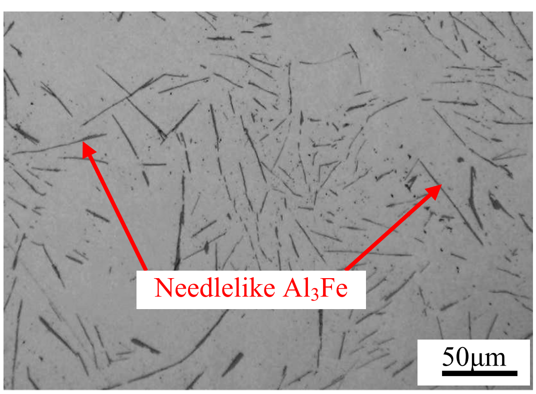

Fig. 3—OM image of as-cast Al-1Fe (wt pct) alloy.

point I arrowed in Figure 2(a). Further, the Fe-bearing phase can be determined as Al_3Fe phase (sometimes it is denoted $Al_{13}Fe_4$),[15,24] based on the corresponding selected area electron diffraction (SAED) (Figure 2(c)). Figure 3 shows the OM image of as-cast Al-1Fe alloy, and it can be found that the shape of Al_3Fe phase is needlelike, similar to that reported for as-cast Al-Fe alloy.[5,6] The average length of the needlelike Al_3Fe phase is about 50 μm, and the Al_3Fe phase in Al-1Fe alloy prepared by rheo-extrusion is much refiner than that prepared by casting.

B. *Mechanical Property of Al-1Fe (Weight Percent) Alloy Prepared by Rheo-Extrusion*

The tensile engineering stress–engineering strain curve of Al-1Fe alloy rod prepared by rheo-extrusion is shown in Figure 4(a), and it is found that the tensile strength and elongation of the Al-1Fe alloy rod are 135 MPa and 30 pct, respectively. In the study of Shi *et al.*,[9] the tensile strength of as-cast Al-1Fe alloy is 85 MPa and the corresponding elongation is 25.5 pct. They also investigated the microstructure and mechanical property of Al-1Fe alloy modified by RE. In the case of the optimal modification effect of RE (0.3 wt pct), Al_3Fe phase could be refined to about 4 μm. However, the tensile strength of as-cast Al-1Fe only increased from 85 to 89 MPa, and the elongation increased from 25.5 to 28.5 pct.[9] The tensile specimens before and after tensile test are shown in Figure 4(c). The comparison of mechanical properties of Al-1Fe alloys prepared by different methods is shown in Figure 4(b). The tensile strength of Al-1Fe alloy was improved by 4.7 pct and elongation was increased by 11.8 pct when Al-1Fe alloy was modified by RE. The tensile strength and elongation of Al-1Fe alloy prepared by rheo-extrusion were 58.8 and 19 pct higher than those of as-cast Al-1Fe alloy. So, Al_3Fe phase can be obviously refined by rheo-extrusion, and the refinement effect is much more significant than that induced by the modification of RE.

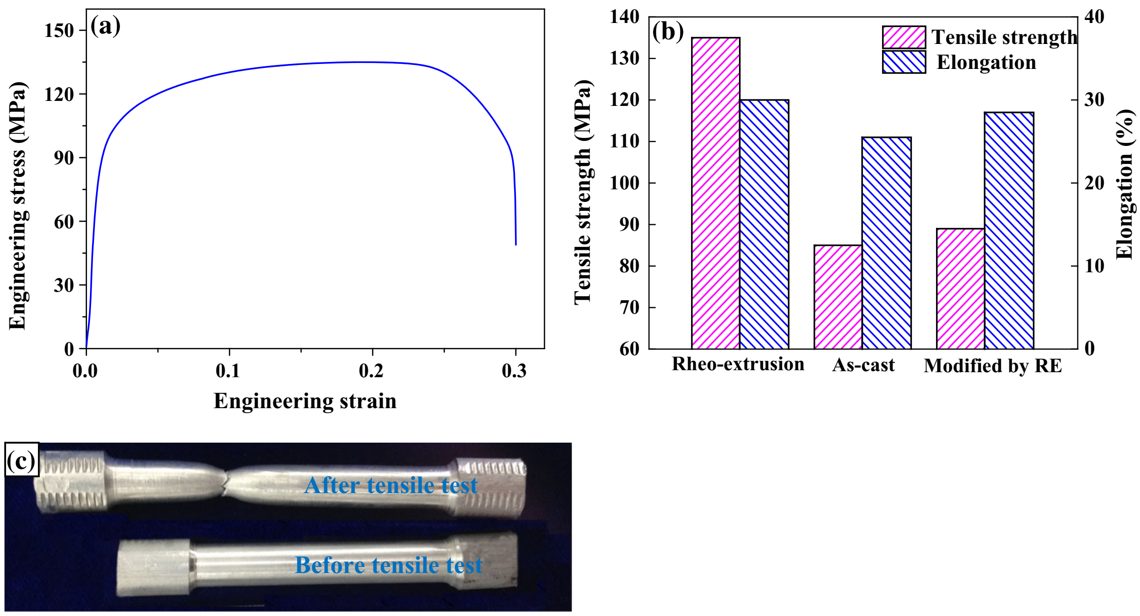

Fig. 4—(*a*) Tensile engineering stress–engineering strain curve of Al-1Fe (wt pct) alloy rod prepared by rheo-extrusion, (*b*) comparison of mechanical properties of Al-1Fe (wt pct) alloys prepared by different methods, and (*c*) tensile specimens before and after tensile testing.

IV. DISCUSSION

To investigate how Al_3Fe phase could be refined to nanoscale during rheo-extrusion, the extrusion machine was stopped and cooled by high-pressure cooling water to take samples for microstructural observation, and the sampling position is shown in Figure 1.

A. *Refinement of Al_3Fe Phase by Shear Deformation in the Wheel Groove*

The microstructure of Al-1Fe alloy at position A is shown in Figure 5(a), and it can be found that the microstructure was composed of α-Al grain as well as eutectic structure. Figure 5(b) shows an SEM image of the eutectic structure in Figure 5(a), and it indicates that the eutectic structure contains bonelike second phase. The EDS analysis in Figure 5(c) shows that the bonelike second phase consists of Al and Fe. The bonelike second phase can be further determined as Al_3Fe phase, according to the X-ray diffraction (XRD) analysis in Figure 5(d). It is well known that Al-1Fe alloy is a hypoeutectic alloy and the eutectic content of Fe is 1.8 wt pct. Primary Al_3Fe phase only existed in eutectic structure formed by eutectic reaction when the content of Fe in residual liquid was enriched to the eutectic content. Further, Figure 5(a) shows that α-Al grain at position A is not elongated, while Figure 5(b) shows that the Al_3Fe phase is not fractured.

At position B, α-Al grain in Al-1Fe alloy was elongated, as shown in Figure 6(a), indicating that the alloy at point B had been completely solidified. Figure 6(b) is the SEM micrograph of Al_3Fe at position B, and it can be found that the average length of bonelike Al_3Fe phase is 2.8 μm and that only a small amount of Al_3Fe phase is broken. When solidified alloy flowed in the roll-shoe gap, shear deformation occurred in the alloy because the friction stresses acted by the rotational extrusion wheel and fixed extrusion shoe were in the opposite directions. The equivalent shear deformation strain of solid metal in the wheel groove has been researched by finite-element model simulation. The study of Wei *et al*. indicated that the equivalent strain of the shear deformation was 0.4 when the diameter of the extrusion wheel was 300 mm,[25] and the report of Cho and Jeong was in full compliance with Wei.[26] However, a different result could be found in the report of Lu *et al*.; they reported that the equivalent strain was 0.53 when the diameter of the extrusion wheel was 500 mm.[27] From the preceding reports, it can be summarized that the equivalent strain of shear deformation in the wheel groove varies with the extrusion wheel's diameter, and the shear deformation in the wheel groove of the DZJ350 rheo-extrusion machine (the diameter of the extrusion wheel is 350 mm) is between 0.4 and 0.53. Further, the alloy is in semisolid state in the front part of the wheel groove, so only little Al_3Fe phase can be fractured by shear deformation in the roll-shoe gap, as shown in Figure 6(b).

B. *Refinement of Al_3Fe Phase by Shear Deformation in Equal Channel Angular Flow*

At the exit of the wheel groove, the flow direction of Al-Fe alloy changed from the tangential direction of the extrusion wheel to the circumferential direction of the extrusion wheel, and the second-stage shear deformation occurred. According to the structure of the experimental device, the second-stage shear deformation can be treated as ECAP; the corresponding Φ (inner corner) is 90 deg and Ψ (outer corner) is 0 deg, so the equivalent strain is 1.155.[28] After the second-stage shear deformation, the amount of fractured Al_3Fe phase increased,

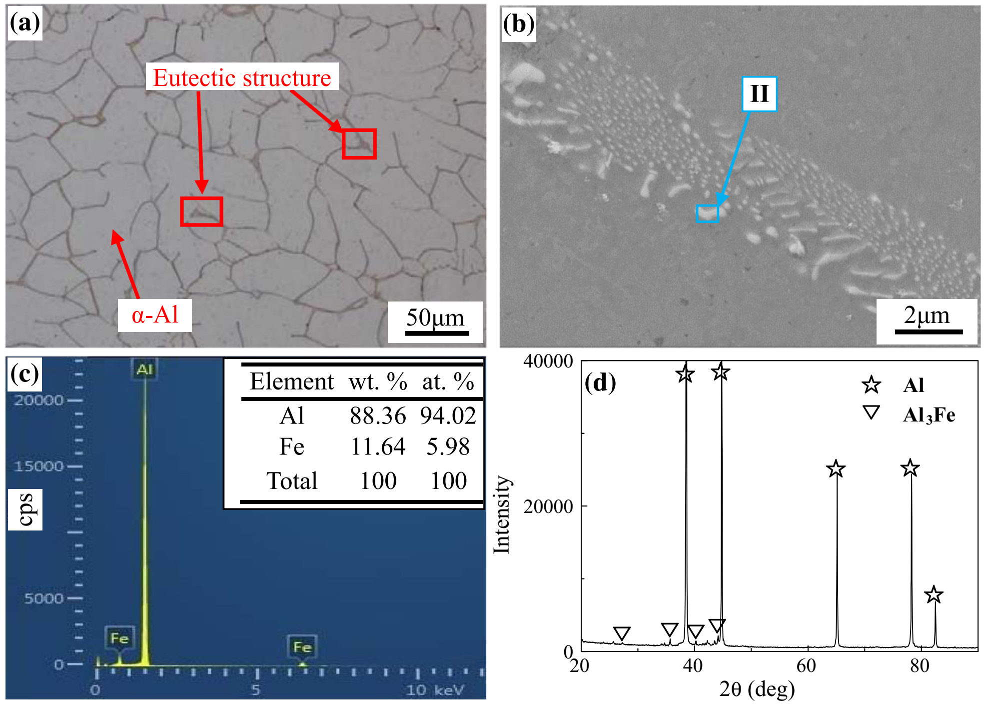

Fig. 5—(*a*) OM micrograph of Al-1Fe (wt pct) alloy at position A. (*b*) SEM image of eutectic structure in (a), showing bonelike second phase distributes along different directions. (*c*) EDS analysis of bonelike second phase at point II in (b). (*d*) XRD analysis of Al-1Fe (wt pct) alloy at position A.

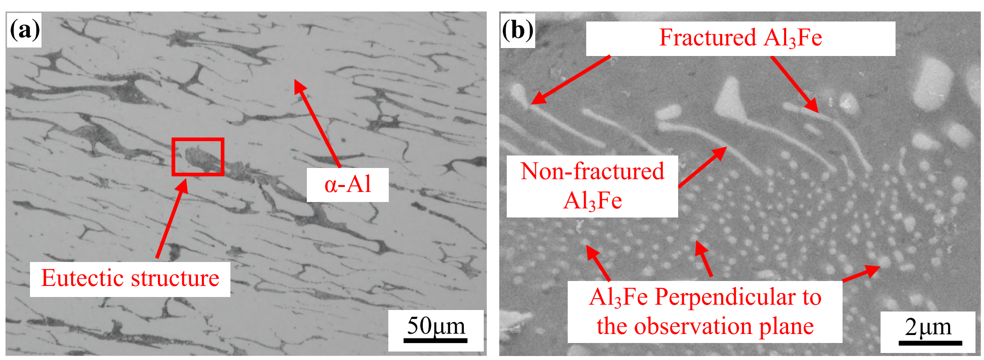

Fig. 6—(*a*) OM micrograph of Al-1Fe (wt pct) alloy at position B. (*b*) SEM micrograph of Al_3Fe phase in (a).

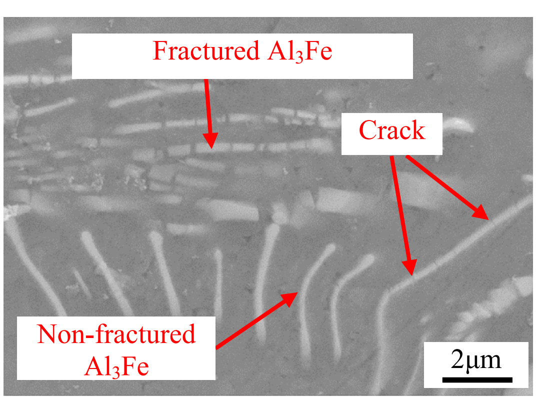

Fig. 7—SEM micrograph of Al_3Fe phase in Al-1Fe (wt pct) alloy at the exit of the roll-shoe gap.

and its average length was 0.8 μm, as shown in Figure 7. The refinement of Al_3Fe phase in Al-Fe alloy by ECAP was reported by Stolyarov,[20] and the bulk Al_3Fe ($Al_{13}Fe_4$) phase was refined from 10 to about 2 μm after one pass ECAP. However, only partial Al_3Fe phase was refined and some bonelike Al_3Fe phase remained (Figure 7).

C. *Refinement of Al_3Fe Phase by Shear Deformation in the Expansion Extrusion Mold*

With the continuous alloy flow, the third-stage shear deformation occurred when Al-Fe alloy flowed into and out of the expansion extrusion mold due to the variation of cavity diameter along the flowing direction. After expansion extrusion, Al_3Fe phase was further refined, and its average length was reduced to 300 nm.

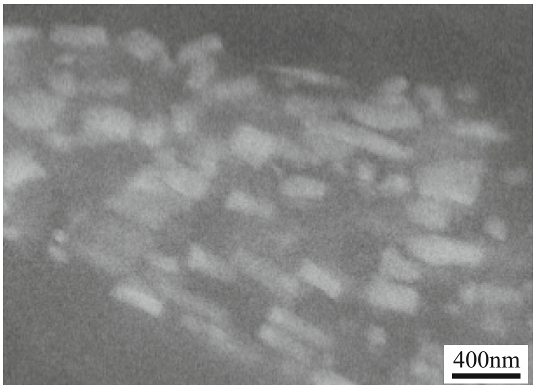

Fig. 8—SEM micrograph of Al_3Fe phase in Al-1Fe (wt pct) in the expansion extrusion mold.

Moreover, there was no nonfractured Al_3Fe, and the length of Al_3Fe phase was homogeneously distributed, as shown in Figure 8. Pardis *et al*.[29] investigated the equivalent strain of expansion extrusion, which could be expressed as $\varepsilon = 4 \ln D_1/D_0$, where D_0 was the diameter of the mold calibrating strap and D_1 was the maximum diameter of the expansion cavity. In our study, D_0 was 10 mm and D_1 was 25 mm, so the equivalent strain suffered by Al-Fe alloy in the expansion extrusion mold was 3.665, which was higher than the equivalent strain of three passes ECAP.

It is generally known that needlelike Al_3Fe phase causes harm to the mechanical property of Al-Fe alloy, but in this article, the mechanical property of Al-Fe alloy is obviously improved when the average length of Al_3Fe phase is reduced to 300 nm. So, it can be noted that Al_3Fe phase also has an obvious strengthening effect when it is refined to nanoscale, and the hated Fe may transfer to a reinforcing particle. Further, rheo-extrusion is a short forming process that has no strict requirement of processing condition and addition of extra elements, and it is suitable for large-scale industrial application. In fact, the distribution uniformity of the nanoscale Al_3Fe phase has the potential to be improved, as do the Al alloys' mechanical properties, which need further study.

V. CONCLUSIONS

In this study, a high-strength Al-1Fe alloy reinforced by nanoscale Al_3Fe phase was obtained by rheo-extrusion. The refining mechanism of Al_3Fe phase in Al-1Fe alloy during rheo-extrusion was investigated by OM, SEM, XRD and TEM, and the mechanical property of the prepared Al-1Fe alloy was measured. The main conclusions are as follows.

1. The average length of Al_3Fe phase in Al-1Fe alloy prepared by rheo-extrusion is 300 nm, which is much more refined than the average length of needlelike Al_3Fe phase in as-cast Al-1Fe alloy (50 μm).
2. In rheo-extrusion, there are three stages of shear deformations, including shear deformation in the wheel groove, equal channel angular flow, and expansion extrusion mold, and the total equivalent strain is higher than 4.82. Al_3Fe phase formed by eutectic reaction was bonelike, but it could be refined by the shear deformation in the wheel groove and equal channel angular flow, and then further refined by the shear deformation in the expansion extrusion mold.
3. The tensile strength and elongation of the Al-1Fe alloy reinforced by nanoscale Al_3Fe phase are 135 MPa and 30 pct, respectively. The tensile strength of Al-1Fe alloy prepared by rheo-extrusion is 58.8 pct higher than that of as-cast Al-1Fe alloy, and the elongation is 19 pct higher than that of as-cast Al-1Fe alloy. The improvements of tensile strength and elongation of Al-1Fe alloy caused by shear deformation in rheo-extrusion are much more significant than the reported improvements induced by RE modification.

ACKNOWLEDGMENTS

The authors are thankful for the support of the National Natural Science Foundation of China under Grant Nos. 51474063 and 51674077 and the Fundamental Research Funds for the Central Universities under Grant No. N150204016.

REFERENCES

1. K.L. Sahoo and C.S. Sivaramakrishnan: *Scripta Mater*., 2003, vol. 99, pp. 253–57.
2. R.G. Guan, Y.F. Shen, Z.Y. Zhao, and X. Wang: *J. Mater. Sci. Technol*., 2016, vol. 33, pp. 215–23.
3. Q.R. Zhao, Z. Qian, X.L. Cui, Y.Y. Wu, and X.F. Liu: *J. Alloys Compd*., 2015, vol. 650, pp. 768–76.
4. P. Zhang, Z.M. Li, B.L. Liu, and W.J. Ding: *J. Mater. Sci. Technol*., 2017, vol. 33, pp. 367–78.
5. P. Moldovan, G. Popescu, and F. Miculescu: *J. Mater. Process. Technol*., 2004, vol. 153, pp. 408–15.
6. S.B. Sun, L.J. Zheng, J.H. Liu, and H. Zhang: *J. Mater. Sci. Technol*., 2017, vol. 33, pp. 389–96.
7. V.V. Tcherdyntsev, S.D. Kaloshkin, D.V. Gunderov, E.A. Afonina, I.G. Brodova, V.V. Stolyarov, Y.V. Baldokhin, E.V. Shelekhov, and I.A. Tomilin: *Mater. Sci. Eng. A*, 2004, vol. 375, pp. 888–93.
8. J.M. Cubero-Sesin and Z. Horita: *Metall. Mater. Trans. A*, 2012, vol. 43A, pp. 5182–92.
9. Z.M. Shi, K. Gao, Y.T. Shi, and Y. Wang: *Mater. Sci. Eng. A*, 2015, vol. 632, pp. 62–71.
10. D. Holland-Moritz, J. Schroers, D.M. Herlach, B. Grushko, and K. Urban: *Acta Mater*., 1998, vol. 46, pp. 1601–15.
11. C.Y. Ban, J.F. Zhang, P. Qian, X. Zhang, Y. Han, and J.Z. Cui: *China Foundry*, 2011, vol. 8, pp. 386–91.
12. S.D. Kaloshkin, V.V. Tcherdyntsev, I.A. Tomilin, D.V. Gunderov, V.V. Stolyarov, Y.V. Baldokhin, I.G. Brodova, and E.V. Shelekhov: *Mater. Trans*., 2002, vol. 43, pp. 2031–38.
13. T. Dorin, N. Stanford, N. Birbilis, and R.K. Gupta: *Corros. Sci*., 2015, vol. 100, pp. 396–403.

14. S.S. Nayak, B.S. Murty, and S.K. Pabi: *J. Mater. Sci.*, 2008, vol. 31, pp. 449–54.
15. L. Li, Y.D. Zhang, C. Esling, H.X. Jiang, Z.H. Zhao, Y.B. Zuo, and J.Z. Cui: *J. Cryst. Growth*, 2012, vol. 339, pp. 61–69.
16. G.F. Mi, C.F. Dong, and D.W. Zhao: *Front. Manuf. Des. Sci.*, 2011, vols. 44–47, pp. 2126–30.
17. Y. Zhang, Y.C. Liu, Y.J. Han, C. Wei, and Z.M. Gao: *J. Alloys Compd.*, 2009, vol. 473, pp. 442–45.
18. O.N. Senkov, F.H. Froes, V.V. Stolyarov, R.Z. Valiev, and J. Liu: *Scripta Mater.*, 1998, vol. 38, pp. 1511–16.
19. J. Hu, J. Teng, X. Ji, X. Kong, F. Jiang, and H. Zhang: *J. Mater. Eng. Perform.*, 2016, vol. 25, pp. 4769–75.
20. V.V. Stolyarov, R. Lapovok, I.G. Brodova, and P.F. Thomson: *Mater. Sci. Eng. A*, 2003, vol. 357, pp. 159–67.
21. Y.F. Shen, R.G. Guan, Z.Y. Zhao, and R.D.K. Misra: *Acta Mater.*, 2015, vol. 100, pp. 247–55.
22. N. Su, R.G. Guan, X. Wang, Y.X. Wang, W.S. Jiang, and H.N. Liu: *J. Alloys Compd.*, 2016, vol. 680, pp. 283–90.
23. Y.X. Wang, R.G. Guan, D.W. Hou, Y. Zhang, W.S. Jiang, and H.N. Liu: *J. Mater. Sci.*, 2017, vol. 52, pp. 1137–48.
24. S. Miyazaki, A. Kawachi, S. Kumai, and A. Sato: *Mater. Sci. Eng. A*, 2005, vol. 400, pp. 294–99.
25. W. Wei, W. Zhang, K.X. Wei, Y. Zhong, G. Cheng, and J. Hu: *Mater. Sci. Eng. A*, 2009, vol. 516, pp. 111–18.
26. J.R. Cho and H.S. Jeong: *J. Mater. Process. Technol.*, 2001, vol. 110, pp. 53–60.
27. J. Lu, N. Saluja, A.L. Riviere, and Y. Zhou: *J. Mater. Process. Technol.*, 1999, vol. 79, pp. 200–12.
28. V.M. Segal: *Mater. Sci. Eng. A*, 1999, vol. 271, pp. 322–33.
29. N. Pardis, B. Talebanpour, R. Ebrahimi, and S. Zomorodian: *Mater. Sci. Eng. A*, 2011, vol. 528, pp. 7537–40.