

# Pembuatan paduan Fe-Cr menggunakan metode ultrasonik = Synthesis of Fe-Cr alloys using ultrasonic method

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## Abstrak

[<b>ABSTRAK</b><br>

Fe-Cr adalah paduan yang memiliki ketahanan temperatur tinggi dan potensial digunakan sebagai interkoneksi pada sel bakar (SOFC=solid oxide fuel cell). Sintesis paduan Fe-Cr terus dikembangkan untuk mendapatkan metode yang efektif, dan efisien. Metode sintesis paduan Fe-Cr yang ada sekarang ini adalah metode peleburan, metalurgi serbuk ataupun metode pemaduan mekanik. Metode-metode tersebut memiliki kelemahan misalnya paduan yang tidak homogen, terdapat oksida, proses panjang dan membutuhkan waktu lama. Untuk meminimumkan permasalahan ini, adalah penting untuk menghasilkan paduan mikro Fe-Cr yang memiliki kestabilan fasa dan sifat mekanis baik. Metode ultrasonik dapat dimanfaatkan untuk sintesis paduan mikro homogen melalui penggunaan gelombang suara ultrasonik. Gelombang suara ultrasonik menghasilkan gelembung-gelembung kavitasi, setiap runtuhannya kavitasi dapat dianggap sebagai reaktor mikro yang mampu menghasilkan temperatur sekitar 4737 oC dan tekanan sekitar 1000 atm dan yang terbentuk dengan sangat cepat, serta menghasilkan gelombang kejut. Dengan demikian metode ultrasonik dapat dimanfaatkan dalam pembuatan paduan mikro Fe-Cr yang homogen serta tanpa oksida dan diharapkan bisa mengatasi kelemahan metode pembuatan paduan berbasis Fe saat ini. Pada penelitian ini telah dilakukan sintesis paduan mikro Fe-Cr dengan metode ultrasonik pada frekuensi 20 kHz dalam cairan toluene. Tahapan yang telah dilakukan adalah perlakuan ultrasonik sebagai variasi waktu terhadap partikel prekursor (Fe, Cr), kemudian terhadap campuran partikel prekursor untuk mendapatkan paduan mikro Fe-Cr. Kemudian dilakukan pembuatan bongkah paduan Fe-Cr dari partikel hasil perlakuan ultrasonik melalui kompaksi tanpa pelumasan dan sintering dalam kapsul kaca kuarsa. Karakterisasi yang dilakukan adalah menggunakan Scanning Electron Microscopy (SEM) terhadap partikel prekursor hasil rekayasa ultrasonik. Untuk partikel campuran prekursor Fe-Cr hasil perlakuan ultrasonik karakterisasi dilakukan menggunakan SEM-EDS (Energy Dispersive Spectroscopy), X-Ray Diffraction (XRD) disertai analisis dengan metode Rietveld, Transmission Electron Microscopy-Selected Area Electron Diffraction (TEM-SAED). Untuk bongkah Fe-Cr hasil konsolidasi dengan menggunakan SEM-EDS, XRD disertai analisis dengan metode Rietveld, pengukuran densitas sebenarnya, pengujian kekerasan Vickers. Efek perlakuan ultrasonik terhadap partikel Fe adalah pengurangan ukuran, penyatuan, dan

aglomerasi. Setelah perlakuan ultrasonik 40 jam terjadi peningkatan ukuran partikel Fe ( $>2\ \mu\text{m}$ ). Terhadap partikel Cr memberikan efek erosi permukaan, pengurangan ukuran dan pemecahan partikel aglomerasi. Partikel Cr aglomerasi terurai sepenuhnya menjadi partikel Cr kecil ( $<2\ \mu\text{m}$ ) setelah 63 jam. Terhadap campuran partikel Fe dan Cr dapat menyatukan partikel kohesif (Fe-Fe, Cr-Cr) dan adhesif (Fe-Cr), terbentuk paduan mikro Fe-Cr seutuhnya (setelah 20 jam) ataupun paduan mikro Fe-Cr sebagian (setelah 50 jam). Pembentukan paduan mikro Fe-Cr diawali pada ukuran partikel Fe ataupun Cr  $<2\ \mu\text{m}$ . Bongkah paduan mikro Fe-Cr yang diperoleh adalah homogen dan tanpa oksida, dengan karakteristik densitas melalui sintering dua tahap yaitu tipe O = 8.655 gr/cm<sup>3</sup>, tipe B=8.179 gr/cm<sup>3</sup>, dan tipe A=8.196 gr/cm<sup>3</sup>, dan melalui proses sintering satu tahap tipe O = 7.678 gr/cm<sup>3</sup>, tipe B=7.587 gr/cm<sup>3</sup>, dan tipe A=7.092 gr/cm<sup>3</sup>. Kekerasan bongkah Fe-Cr terbesar melalui sintering satu tahap yaitu 88 VHN adalah tipe B, sementara terbesar dua tahap yaitu 81 VHN adalah tipe A. Proses perlakuan ultrasonik memberikan dampak positif baik dari sisi waktu proses maupun kualitas hasil paduan Fe-Cr. Dengan demikian metode ultrasonik bisa diandalkan sebagai alternatif dalam pembuatan paduan berbasis Fe untuk mengatasi kendala homogenisasi dan oksidasi yang dihadapi pada metode saat ini.;

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<b>ABSTRACT</b><br>

Fe-Cr alloys have the potential for use as an interconnection material for solid oxide fuel cell (SOFC) due to its being resistance to high temperatures. Synthesis methods of Fe-Cr alloy continue to be developed in order to obtain a method that is both effective and efficient. Presently, the synthesis of Fe-Cr alloys include the casting, the powder metallurgy, and the mechanical alloying method. These methods have several drawbacks such as inhomogeneity in the resulting products, oxidation, and require a very time-consuming process to accomplish. In order to minimize this problem, it is important to produce Fe-Cr microalloys. Fe-Cr microalloys exhibit phase stability and good mechanical properties. Ultrasonic methods can be used in the synthesis of homogeneous microalloys by employing the ultrasonic sound waves. Ultrasonic sound waves will generate cavitation bubbles. Any cavitation collapse can be considered as a micro reactor in which a temperature of about 4737 oC and a pressure of about 1000 atm a very rapidly created, thereby generating a shock wave. Thus, the ultrasonic method can be used in producing homogeneous and free-oxide Fe-Cr microalloys and can be expected to overcome the limitations imposed by the current methods. In this work the formation of Fe-Cr microalloys by ultrasonic treatment at a frequency of 20 kHz in toluene liquid is presented. In the synthesis procedure, the procedure steps followed were: (1) the treatment of precursor particles (Fe, Cr) through ultrasonic method with a time-variation, followed by (2) the same time-varying ultrasonic treatment on the admixture of these specially prepared precursor particles in order to obtain the Fe-Cr microalloys, and (3) finally, the lubricantless

compaction method was employed on these precursor particles admixture followed by sintering process inside quartz tubes to obtain a bulk of Fe-Cr alloy. Observations of the specially prepared precursor particles using ultrasonic technique were carried out by scanning electron microscopy (SEM) method. Observation of the precursor mixture of Fe-Cr particles mixture treated ultrasonically was performed using a SEM-EDS (energy-dispersive spectroscopy) apparatus, a X-Ray diffractometer and accompanied by the Rietveld analysis method, and transmission electron microscopy (TEM)-selected area electron diffraction (SAED) apparatus. The bulk of Fe-Cr alloy were observed using SEMEDS, XRD accompanied by analysis by the Rietveld method, true density measurement, and Vickers microhardness testing. Ultrasonic treatment has caused Fe particles to form agglomerations, an interparticles neck formation, and a fusing among the particles. The size of the Fe particles increased ( $>2\ \mu\text{m}$ ) after 40 hours treatment. The agglomerated Cr particles experienced fragmentation, surface erosion, and reduction of particle size. The agglomerated Cr particles fully disintegrated into Cr microparticles ( $<2\ \mu\text{m}$ ) after 63 hours treatment. The mixture of Fe-Cr forming cohesive (Fe-Fe, Cr-Cr) and adhesive (Fe-Cr) particles, forming completely (after ultrasonic treatment for 20 hours) and partially (after ultrasonic treatment for 50 hours) Fe-Cr microalloys. The complete formation of Fe-Cr microalloy was possible with an equal particle size of the precursor Fe and Cr (approximately  $<2\ \mu\text{m}$ ). The bulk of Fe-Cr alloy results are homogenous and oxide-free. For two-step sintering, its density (in  $\text{gr}/\text{cm}^3$  unit) is 8.655 for type O, is 8.179 for type B, and is 8.196 for type A, and for one-step sintering its density is 7.678 for type O, is 7.587 for type B, and is 7.092 for type A. The greatest microhardness number of 88 VHN is of type B (one-step sintering), and of 81 VHN is of type A (two-step sintering). The ultrasonic treatment process has a positive impact, with respect to both of quality and time-consumption to finish the Fe-Cr alloying process. Therefore the ultrasonic method can be relied upon as an alternative method in the production of Fe-based alloys to solve problems in homogenization and oxidation encountered in current methods; Fe-Cr alloys have the potential for use as an interconnection material for solid oxide fuel cell (SOFC) due to its being resistance to high temperatures. Synthesis methods of Fe-Cr alloy continue to be developed in order to obtain a method that is both effective and efficient. Presently, the synthesis of Fe-Cr alloys include the casting, the powder metallurgy, and the mechanical alloying method. These methods have several drawbacks such as inhomogeneity in the resulting products, oxidation, and require a very time-consuming process to accomplish. In order to minimize this problem, it is important to produce Fe-Cr microalloys. Fe-Cr microalloys exhibit phase stability and good mechanical properties. Ultrasonic methods can be used in the synthesis of homogeneous microalloys by employing the ultrasonic sound waves. Ultrasonic sound waves will generate cavitation bubbles. Any cavitation collapse can be considered as a micro reactor in which a

temperature of about 4737 °C and a pressure of about 1000 atm a very rapidly created, thereby generating a shock wave. Thus, the ultrasonic method can be used in producing homogeneous and free-oxide Fe-Cr microalloys and can be expected to overcome the limitations imposed by the current methods. In this work the formation of Fe-Cr microalloys by ultrasonic treatment at a frequency of 20 kHz in toluene liquid is presented. In the synthesis procedure, the procedure steps followed were: (1) the treatment of precursor particles (Fe, Cr) through ultrasonic method with a time-variation, followed by (2) the same time-varying ultrasonic treatment on the admixture of these specially prepared precursor particles in order to obtain the Fe-Cr microalloys, and (3) finally, the lubricantless compaction method was employed on these precursor particles admixture followed by sintering process inside quartz tubes to obtain a bulk of Fe-Cr alloy. Observations of the specially prepared precursor particles using ultrasonic technique were carried out by scanning electron microscopy (SEM) method. Observation of the precursor mixture of Fe-Cr particles mixture treated ultrasonically was performed using a SEM-EDS (energy-dispersive spectroscopy) apparatus, a X-Ray diffractometer and accompanied by the Rietveld analysis method, and transmission electron microscopy (TEM)-selected area electron diffraction (SAED) apparatus. The bulk of Fe-Cr alloy were observed using SEMEDS, XRD accompanied by analysis by the Rietveld method, true density measurement, and Vickers microhardness testing. Ultrasonic treatment has caused Fe particles to form agglomerations, an interparticles neck formation, and a fusing among the particles. The size of the Fe particles increased ( $>2\ \mu\text{m}$ ) after 40 hours treatment. The agglomerated Cr particles experienced fragmentation, surface erosion, and reduction of particle size. The agglomerated Cr particles fully disintegrated into Cr microparticles ( $<2\ \mu\text{m}$ ) after 63 hours treatment. The mixture of Fe-Cr forming cohesive (Fe-Fe, Cr-Cr) and adhesive (Fe-Cr) particles, forming completely (after ultrasonic treatment for 20 hours) and partially (after ultrasonic treatment for 50 hours) Fe-Cr microalloys. The complete formation of Fe-Cr microalloy was possible with an equal particle size of the precursor Fe and Cr (approximately  $<2\ \mu\text{m}$ ). The bulk of Fe-Cr alloy results are homogenous and oxide-free. For two-step sintering, its density (in  $\text{gr}/\text{cm}^3$  unit) is 8.655 for type O, is 8.179 for type B, and is 8.196 for type A, and for one-step sintering its density is 7.678 for type O, is 7.587 for type B, and is 7.092 for type A. The greatest microhardness number of 88 VHN is of type B (one-step sintering), and of 81 VHN is of type A (two-step sintering). The ultrasonic treatment process has a positive impact, with respect to both of quality and time-consumption to finish the Fe-Cr alloying process. Therefore the ultrasonic method can be relied upon as an alternative method in the production of Fe-based alloys to solve problems in homogenization and oxidation encountered in current methods, Fe-Cr alloys have the potential for use as an interconnection material for solid oxide fuel cell (SOFC) due to its being resistance to high temperatures.

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