

Recovery of Aluminium 319.1 Alloy(S) From Metal Turning Scrap

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Abstract

The concern of this work was to recover the standard Al 319.1 alloy as specified; Al-Cu-Zn and Mg from turning scrap by hydrometallurgy method. The activity process utilizes H₂O₂ in ammonia/ammonium carbonate in order to dissolve copper by selection. Tin and lead leached with hydrochloric acid in hot conditions. Lead chloride precipitates on cooling. Leaching reborn into carbonate by leach with soda ash solution. After analyses, the exact volume of solution that contain the required weight of the designated metal was mixed. Metals of tin, copper and lead recovered from the leach merchandise by chemical reduction victimization reducer hydrazine hydrate or ascorbic acid. The metals particles so obtained were filtered, compacted by pressing and melted under a carbon/alkali salt flux. Parameters poignant the recovery potency of the investigated method, like time, temperature, H₂O₂, hydrazine hydrate concentration and stoichiometric ratio 1: 1. 1-1.5 of the acid: solid turnings have been investigated. Results obtained show that melting of the turnings at temperature up to 1400 °C didn't recover the commonplace alloy due to thermal volatilization of tin and escape of lead into the scum. The hydrometallurgical process is vital to sick metals from the turning's scrap oxide of the metals originally gift within the scrap. Metals getting into the scum were recovered within the same manner. Free metals were recovered from the leached products by reduction with the aqueous solution of hydrazine hydrate. The pressed metal powder was sintered at 550 °C for 10 minutes. The activation energy of the latter method amounts to 83.6, 12.7 and 51.73 kJ/mol for copper, lead and tin respectively. the most recovery potency of the recommended methodology amounts to 98.7%. A preliminary economic analysis suggests that the method may be feasible for wailing and structure applications.

Keywords: Al 319.1 alloy, waste, low temperature synthesis, Al-Cu-Zn-N- Mg alloy Key Words Al 319.1 alloy, waste, low temperature synthesis, Al-Cu-Zn-N- Mg alloy

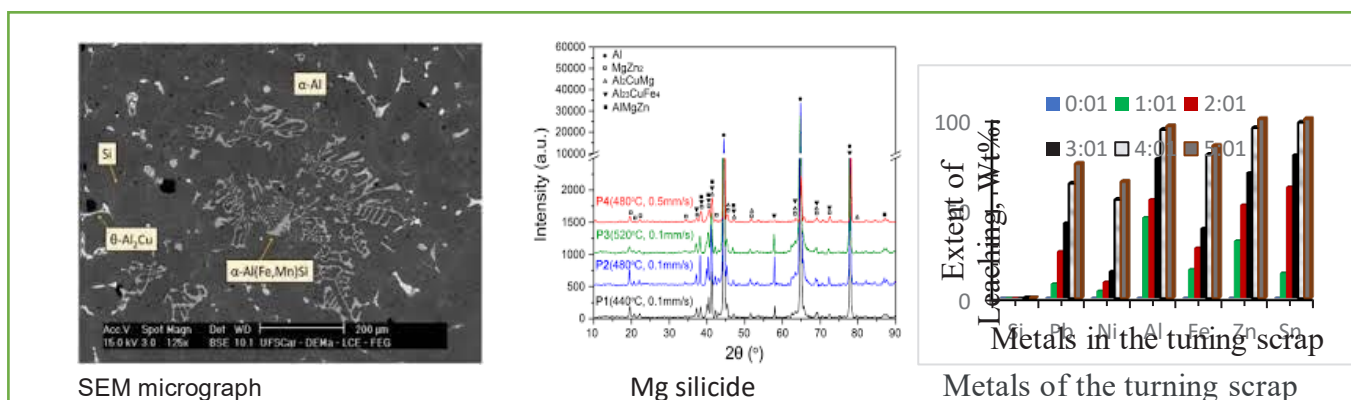


Figure 1: The Graphical abstract

Introduction

The Al alloy 319.1 is highly recommended in structural application where lightweight or corrosion resistance is required. Alloys, composed mostly of aluminium, display importance in aerospace manufacturing. The alloy is used for the production of electronic and microelectronic components and radar construction. The new patent-pending aluminium alloy from Kobe Steel is found to employ a sprig-forming process and has been said to be the strongest aluminium alloy available [1]. Yih and Wang 1979 reported that the hydrometallurgy and pyrometallurgy processes were successful methods to recover valuable metals from the WC scrap [2]. The European Commission showed that several member States proposed that there should be separate customs codes for scrap assortment [3]. This could allow all enforcement personnel to figure with the identical parameters and reduce uncertainty scrap considering as waste. It is assessed in line with NACE codes 2011 and/or CN trade codes (CN Codes, 2011).

1. Katiyara et al [4]. claimed that the recovery of W and Co metals was disbursed by either hydro or pyrometallurgical methods or a combination of them. Direct leaching of tungsten scrap in concentrated acid/alkali solutions has also been investigated. Different value-added materials like ammonium para tungstate, oxyacid etc. were produced.
2. An electrochemical route has emerged as a horny method because it should be one step dissolution process consuming very low energy. However, passivation has been reported to impede the dissolution rate and hence, some additives have also been tried for continuous dissolution. The zinc melt process method was disclosed by Edtmaier, 2005 whereby molten zinc rapidly alloyed with cobalt binder and embrittle the cemented carbide [5]. Both the zinc and cobalt were removed by treatment with acid, and only tungsten carbide could be reused directly. Removal of the zinc from Zn-Co alloy by distillation was invented by Barnard et al. (1971) [6].

In the hydrometallurgical route, the scrap is immersed in a leaching solution to dissolve the matrix or binder material so the inactive residue of the tungsten carbide was formed. The residue of carbide material was ground and a powder material is reused for the preparation of cement carbides. The advantage of that process is that metal carbide is often directly produced and thus the number of operations involved became less. The chlorination method was also reported by Jonsson 1971, where scrap material was subjected to chlorination at high temperature in an exceeding chlorine atmosphere to form metal chloride [7]. The foremost stages of the metal recycling process are as follows: collection, sorting and shredding.

Shredding is completed to plug the melting process. Small shredded metals have an oversized surface to volume ratio, melting and purification (Lablanc [8]). The author reported the influence of the temperature of the melt overheating and pouring on the quality of aluminium alloy castings. The proper melting temperature is 880–890°C. Chen et al [9]. showed that higher temperature or high billet temperature reduced the grain size and increased the volume fraction of Al₂₃CuFe₄ and Al-Mg-Zn alloys Laverty et al [10]. showed that the U.S. Bureau of Mines treated the mixed and contaminated superalloy scrap (by pyrometallurgical and hydrometallurgical methods) to separate and recover metal values. Results obtained by leaching Zn or atomized scrap with HCl at 950 °C and 50 psig whereby approximately 98 % of the Al, Co, Cr, Cu, Fe, Mn, Ni, and Zn dissolved while rejecting over 98 % of Mo, Nb, Ta, Ti, W, and Zr as an insoluble refractory residue. Chlorine successfully substituted HCl to leach Zn-treated scrap but was unsuccessful for leaching atomized scrap. The leaching solution was treated by pH adjustment and hydrothermal precipitation at 2000 °C for 4 h to remove Al, Cr, Fe, and other contaminants as a filterable precipitate. Recovery of Co and Ni would be accomplished by solvent extraction and

Electrowinning. Chromium recovery as a ferroalloy was demonstrated. Mashhadi et al [11], investigated the recovery of aluminium alloy turning scrap via cold pressing and melting with salt flux. The Authors showed that the major elements of all products were in the range of AA 336 aluminium alloy ingot. Melting cold-pressed specimens in salt flux and in molten aluminium alloy produced samples with mechanical properties almost equal to the samples obtained from melting ingot. Commodity evolution [12, 13], with independent market research and surveys, reported open and big data provides prices of more than 250 types of ferrous, non-ferrous scrap and non-ferrous metal alloys, classified in international areas. Worldwide metal scrap has been indexed as: DIN / ISO / EN / IS / ASME in both Metric & Imperial Inch sizes [14]. The scrap material obtained from the El-Maady Engineering company (military factory 54), 30 kg of the scrap turnings left after turning machines. Table 1 shows the content of the metal of the scrap.

Experimental

The metals scrap characteristics

The scrap material obtained from the El-Maady Engineering company (military factory 54), 30 kg of the scrap turnings left after turning machines. Table 1 shows the content of the metal of the scrap.

Table 1: The content of the metals scrap characteristics.

Sampling conditions	Sample#1, 4. Kg	Sample #2, 5 kg	Sample #3 6.5 kg	Sample #4 7.0 kg	Sample #4 7.5 kg	all samples, 30.0 kg
Sampling date	Metal, Wt. %					
1-Mon. Mar. 2, 2020	Al, 25.4	26.05	34.24	18.55	21.81	7.4768 kg
	Si 6; 2	7.1	Si 6.08;	8.75	5.23	1.9914 kg
2- Mon. Mar. 16, 2020	Cu 3.5	20.8	Cu 8.5	27.2	30.61	1.904 kg
	Ni, 8.2	5.1	Ni, 8.2	7.62	3.22	4.02747 kg
3- Mon. April 6, 2020	Fe, 46.1	17.24	Fe, 29.1	29.15	33.47	9.144 kg
	Pb, 4.2	9.71	Pb, 5.2	3.33	--	1.2746 kg
4- Mon. April 50, 2020	Zn, 3.8	6.40	Zn, 4.8	6.67	4.25	1.5696 kg
	Sn, 1.9	7.46	Sn, 1.9	2.06	1.09	0.7985 kg
	Others,0.7	0.04	0.98	0.00	0.32	1.838 kg

Table 2. shows classification of consolidated and innovative sorting methods showing the physical parameter and the desired separation; the developed technologies as reported for each method. Data elaborated from [15].

Table 2: classification of consolidated and innovative sorting methods

Method	Separator Type	Physical Parameter	Desired Separation	Technology
Consolidated methods	magnetic separator	magnetic susceptibility	ferrous fraction, nickel-based alloy	magnetic drum, overhead belt magnet
	air separator	mass	low density as paper, foam plastic	vertical zig-zag, air table, elutriator, air knives
	eddy current	conductivity	non-metal, and metal types	eddy current system, electromagnetic system
	dense media	density	non-metal and metal types	soak float, wet jig
	hand sorting	aspect	metal types and wrought-casting alloys	manual operation
	thermal	melting point	wrought-casting alloys	hot crush
Innovative methods	elemental composition	vapor phase, plasma, x-ray energy, γ -ray energy	alloy type	LIBS, XRF, PGNAA *
	image analysis	color and shape	alloy type	color, etch, 3D shape
	transmission	atomic number	alloy type	XRT *

* XRF: X-ray fluorescence, LIBS: laser-induced breakdown spectroscopy, PGNAA: prompt gamma neutron, activation analysis, XRT: (X-ray transmission).

Figure 2: shows two photographs of the turning scrap.



Stainless steel scrap

Figure 2: Photographs of the metal turnings scrap.



Ferrous and nonferrous scrap

Scrap sorting

Scrap traders, at the commercial level, may sort metal scrap by various methods including magnets, or observe the metal's colour or weight to determine the metal type. For example, aluminium appeared silvery and light, copper looks red whereas brass is yellow. The scrappers will be segregating clean metal from the dirty one to improve the value of their selling scrap. Sorting of the ferrous metals from non-ferrous metals is an important step in that process.

Dry grinding of the scrap

Usually, with dry grinding in the machine, the material will collide and hit other particles in a closed area until it has broken into the required size. Dry milling can thus make large-size particles reach micron size. But if smaller nano-meter sizes are targeted, the only way to achieve this goal is by wet grinding.

Figure 3: shows a Laboratory scale fluidized bed jet mill. The ALPA fine grinding MQW Fluidized Bed Jet Mill (Single Rotor / Multiple Rotor ring mill).



Figure 3a: A pilot-scale Grinding mill



Figure 3b: The powdered turning scrap

The chemicals

The chemicals used in this work were of pure grade, Table 3 shows the properties of the chemicals used.

Table 3: Properties of the chemicals,

chemical	Source	Purpose	Property
Nitric acid H2SO4 HCl	SP.GR.1.18 (AR) Min. assay 36 % Fuming 69 % H2SO4 95-97% Extra pure SP.GR.1.18 (AR)	Leaching Process	ADWIC Riedel- de Hein ADWIC
Ca CO3 EJSF2 Ca oxide	Green Egypt Sigma Aldrich	Synthesis process	99.3, 1.6 um 3.34 g/cm3, 1.57 um
NaOH Ammonium hydroxide	United Co. for chemicals & Med. Preparations	United Co. for chemicals & Med. Prepa- rations	reagent for analysis 25 % Pure reagent for analysis
AgNO3 (Silver Nitrate)		Chloride ion determination	Pure reagent for analysis
distilled water		Chemical reactions	
Tap water		Other purposes	

Synthesis of magnesium silicide

Magnesium silicide was prepared to have EC number 245-254- and the crystal structure as shown in Fig. 3. The Mg Silicide was synthesised according to the method given in Wikipedia and others [12, 15-19]. by heating silicon dioxide, SiO₂, with excess magnesium metal apart of atmospheric oxygen.. The process first forms silicon metal and magnesium oxide,:



Mg₂Si formed from the reaction of the remaining magnesium with the silicon:



These reactions proceed exothermically [4] even explosively. Fig 4 shows the structure of Mg silicide so formed. Mg₂Si crystallizes in the antiferroite structure. In the face-centred cubic lattice, Si canters occupy the corners and face-centred positions of the unit cell and Mg centres occupy eight tetrahedral sites in the interior of the unit cell. The coordination numbers of Si and Mg are eight and four, respectively [20].

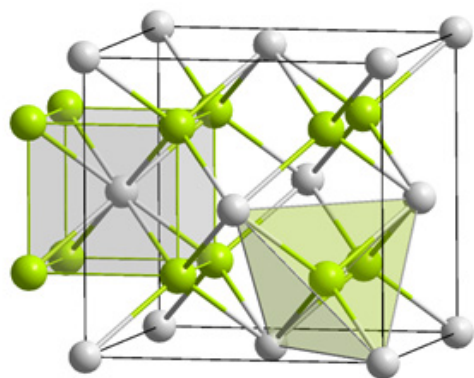


Figure 4: The structure of Mg silicide

Method of the preparation of the Al 319.1 alloy

The scrap collective sample was washed with kerosene, rinsed with sodium oleate solution, distilled water and dried. It was then milled to pass a mesh size of 60 microns and homogenized. The ground sample leached in H₂O₂ in 30% ammonia/ammonium bicarbonate (1:1) to leach copper selectively. The remaining metals then leached in 3M H₂SO₄ acid at 85 °C for different times up to 6 h to leach the Al, Ni, Fe, Mn, Mg and Zn metals. The soluble metals solution was filtered and adjusted for a pH value of 6.5-7 prior to checking by spectrophotometric analyses. The volume of the metal solutions that contain the exact amount of the metals required to form the 319.1 alloys separated with the help of a separating funnel. Metals in shortage completed by addition of a primary salt solution. Magnesium and silicon adjusted by adding the proper amount of the synthesised Mg silicide. The mix reduced to the respective metals using 1 M ascorbic acid. The metals particles so formed filtered, washed, dried and pressed to solid billets under a pressing force of 500 MPa. The billets were annealed at 850 °C- 1100°C for 10 h for improving the physical and mechanical properties of the alloy, eliminating inner stresses, reducing structural hardness, reducing structural hardness, greater structural uniformity, improved ductility and fracture prevention [20], greater structural uniformity, improved ductility and fracture prevention [21]. Fig. 5 shows a sequential processing diagram for the preparation of valuable 319.1 alloy from metal turning scrap. The targeted 319.1 alloy has the following composition as given in table 4.

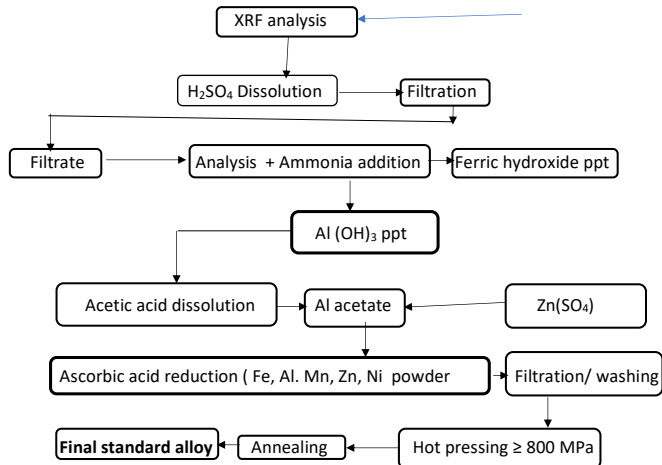


Figure 5: Sequential processing diagram for preparation of valuable 319.1 alloys from metal turning scrap.

Table 4: Composition of the 319.1 alloy

Element	Wt. pct
Aluminium, Al	≈90 %
Copper, Cu	3.5 %
Iron, Fe	<0.80 %
Magnesium, Mg	<0.10 %
Manganese, Mn	<0.50 %
Nickel, Ni	<0.35 %
Other, total	<0.50 %
Silicon, Si	5.5 %
Zinc, Zn	<1.0 %

Results

Fig. 1 shows the graphical abstract containing the SEM micrograph of the Al alloy 319.1. Fig. 2 shows a photograph of the metal's turning scrap. Fig 4 shows the structure of Mg silicide. Silicon occupies the centres and the corners of the face-centered cubic lattice, and Mg centres occupy eight tetrahedral sites in the interior of the unit cell. Fig. 6 shows the effect of particle size of the shredded scrap on the cumulative weight pct of the magnetically-captured scrap subjected to a magnetic field of 5 k Gauss. It is seen that the captured weight of metals in the turning decreases in the order ferromagnetic metals, paramagnetic and diamagnetic metals. The increase in the particle size of the shredded scrap causes a corresponding increase in the weight of the captured metal. The magnetic separation decreases in the order ferromagnetic, diamagnetic and paramagnetic metals.

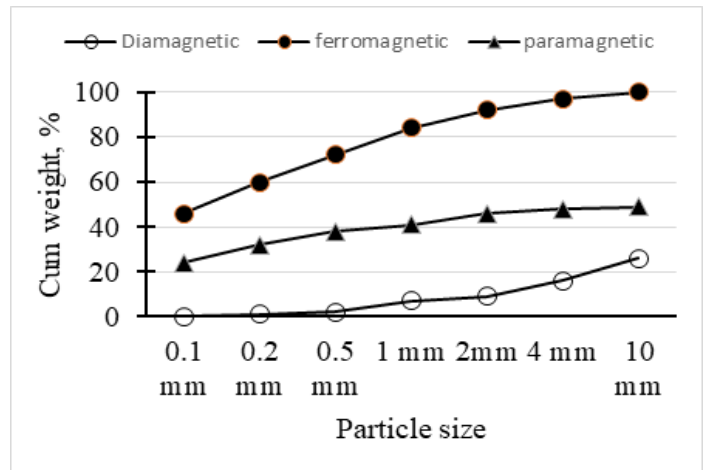


Figure 6: Effect of particle size of the shredded turnings scrap on the cumulative weight pct subjected to 5 k Gauss magnetic field.

Fig. 7 shows the extent of selectively removing copper metal by leaching in H₂O₂ (20 %) in 1 M solution made of ammonia/ ammonium bicarbonate (1:1 mole ratio) at temperatures up to 90 °C. It can be seen that the extent of leaching copper increases with the increase in time and temperature.

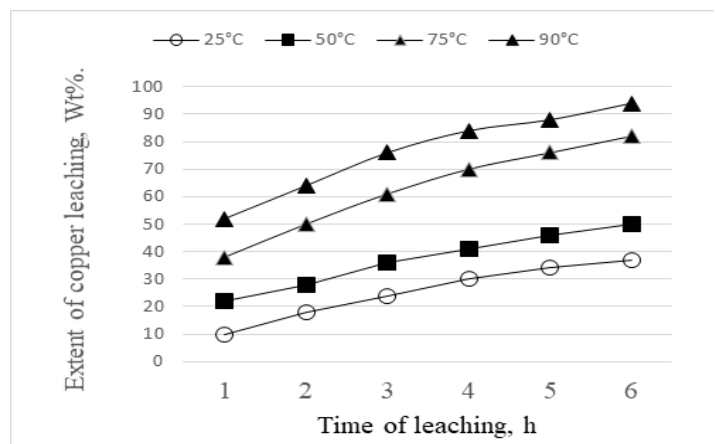


Figure 7: Effect of time and temperature of the H₂O₂ (20%) in 1 M ammonia/ammonium carbonate on the extent of leaching copper from the turning scrap.

Fig. 8 shows the effect of different concentration of HCl acid at 85 °C on the extent of leaching of metals scrap remaining after copper separation. It can be seen that complete leaching of the metals in scrap takes place at 90 °C and after 6 hours of treatment.

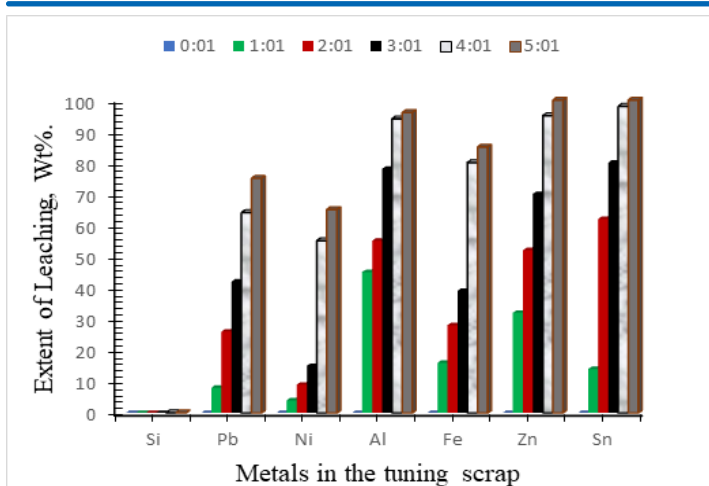


Figure 8: Effect of different concentration of HCl acid : solid mass ratio at 85 °C on the extent of leaching of metals in the scrap remaining after removal of copper-

Fig. 7 shows the effect of different concentration of HCl acid : solid mass ratio at 85 °C on the extent of leaching of metals in the scrap remaining after removal of copper-It is seen that the extent of leaching decreases in the order, Sn, Zn, Al, Fe, and nickel. Other metal of Si doesn't dissolve. Fig. 9 shows the rate of dissolution of the remaining metals after ammonia leaching in 3M HCl at room temperature. It can be seen that magnesium readily dissolves in the HCl acid whereas other metals display a rate of dissolution decreasing in the order Fe, Al, Mn and Ni. Silicon is resistant to the acid attack.

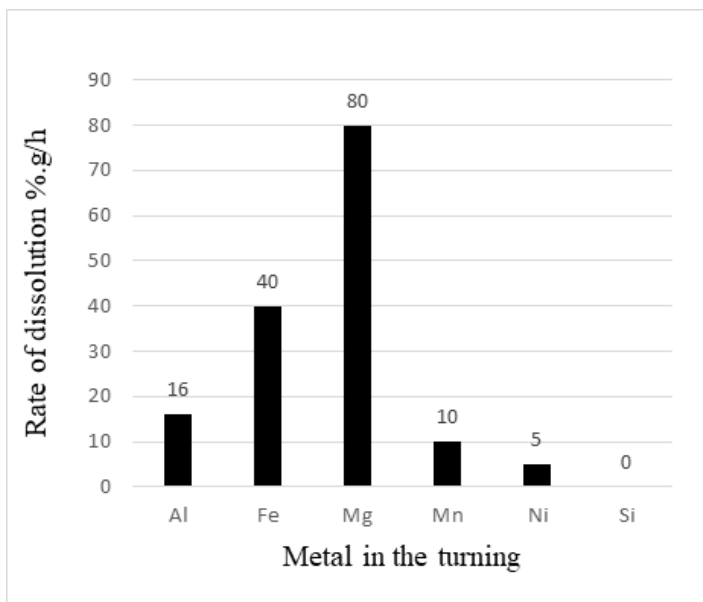


Figure 9: The rate of dissolution of some metals in the turning in 3 M HCl at room temperature.

Discussion

Aluminium alloy 319.1 is of interest for researchers in wailing and structure applications on basis of its outstanding mechanical and chemical properties. The input metals, in this study, are the turning

scrap recurring in the machining workshops of metals, in general, and in the military industry in particular. The turning scrap sample contains different metals such as Al-Zn, copper, stainless steel and other metal alloys. Table 1 shows the metal content of the worked turning scrap sample collected over one month of the year 2020. Because steel alloy(s) may form the major ingredient and weight pct in the turning scrap, it becomes legitimate to remove steel and other ferromagnetic species from the input scrap before start working of this study as far as the Al 319.1 alloy is the target alloy. Table 2 shows the common ferromagnetic and paramagnetic minerals.

Table 2: The common ferromagnetic and paramagnetic minerals

	Mineral	Formula	Field Strength (kG)
Ferromagnetic	Magnetite	FeO	1
	Pyrrhotite	FeS	0.5 - 4
Paramagnetic	Ilmenite	FeOTiO ₂	8 - 16
	Siderite	FeCO ₃	9 - 18
	Chromite	CrO ₃	10 - 16
	Hematite	Fe ₂ O ₃	12 - 18
	Wolframite	(Fe-Mn)WO ₄	12 - 18
	Tourmaline	B ₂ SiO	16 - 20

The direct pyro metallurgy method of turning doesn't score the goal due to the wide variation of the melting point of the elements. For example, zinc has a low melting point of 420 °C and aluminium melts at 660 °C. Carbon steel and nickel melting point amount to 1425 °C and 1433°C respectively (HMP). Low melting point elements (LMP) would volatilize in case heating performed at the temperature level of the high melting point elements. Such a phenomenon would cause a significant loss in the LMP elements. Thermal treatment at the low temperature has proved inappropriate to attain the required composition and the adequate homogeneity of the elements that form the 3129.1 alloys. Because of these technical reasons, the pyrometallurgy method has been set aside and excluded. The hydrometallurgy method, therefore, would be the unique way to score the goal. It involves thoroughly metals composition and mixing of the leached solutions of the respective metals followed by chemical reduction of the ionic state of the elements of concern using a powerful reducing agent such as hydrazine hydrate or ascorbic acid. Copper in the scrapped material is removed to manage the required inclusion in the 31.9.1 alloy. The metal copper was dissolved in hydrogen peroxide in 30% ammonia and ammonium carbonate solution. Fig.5 shows the leaching extent as affected by time at room temperature. It is seen that the extent of leaching increases with an increase in time. Such effect is reasonable in the light of the mechanism of copper dissolution in the alkaline medium as follows. H₂O₂ dissociates to form H₃O and O₂H ions 23.



$\Delta H = + 8.6$ kcal. Calculated from the slope of the Arrhenius plot.

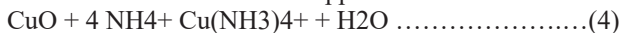
The O_2H ions then dissociate to O and OH group"



The liberated O reacts with Cu to form CuO

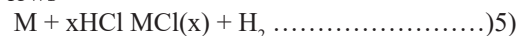


The activation energy of reaction 3 amounts to 40 kJ/mole at an oxygen pressure of 0.1- 1.0 KPa [24]. It is that CuO reacts with ammonium radical to form copper tetrammonia



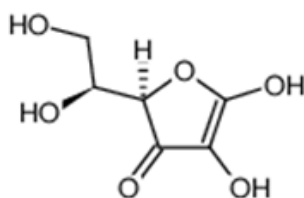
Reaction 3 is the rate-determining step.

Other metals in the turning undissolved in this alkaline reagent. After filtration, copper goes into the solution. The solid residue is leached by 3 M HCl solution to produce soluble chloride as follows



where x equals ≥ 1 .

The metals in the chloride solution were analysed and the required mole was adjusted either by partial precipitation of the metal in excess to form an insoluble compound or by addition of a freshly prepared solution from a primary salt of the metal of concern. The calculated volume of the metal's chloride solution was then mixed with copper tetrammonia and magnesium silicide and thoroughly stirred. Metal ions in the global solution were reduced chemically with hydrazine hydrate or ascorbic acid as follows. Ascorbic acid has the following structure



whereby the (...H) is the active reducing atom

The reaction with MCl_2 to give free metal particles takes place according to



M denotes the metal in the solution.

Similarly, hydrazine hydrate reduces the metal ions according to

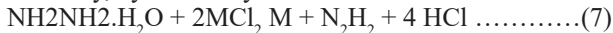


Fig. 10 shows scan images of copper, Al, Zn, Fe, Mn and nickel nanoparticles obtained by chemical reduction of their respective salt solution using 1M ascorbic acid at room temperature. Magnesium and silicon content were managed by the addition of the freshly prepared magnesium silicide. Fig 3 shows the structure of Mg silicide. Silicon occupies the centres and the corners of the face-centered cubic lattice. and Mg centres occupy eight tetrahedral sites in the interior of the unit cell. Fig. 2 shows a photograph of the metal's turnings scrap Fig. 10 shows SEM images of some metals obtained by reduction of their salts with ascorbic acid 1 M solution.

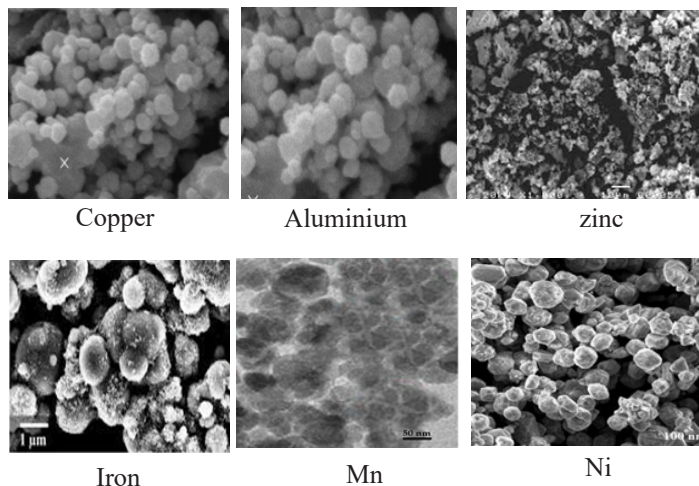


Figure 10: SEM images of some metals obtained by reduction of their salts with ascorbic acid 1 M solution.

Fig. 11 a shows the image of the prepared discs of the 319.1 alloy. The discs were prepared of the filtered metal particles and compacted via compressing in a tool steel die 20 mm inner diameter under a compressive force of 500 kPa. The green discs were annealed for ≥ 10 h at 850 °C – 1100 °C to get rid of the internal stresses and removal of any voids.

Fig. 11b shows photograph of some shapes of the prepared Al 319.1 alloy.



Figure 11a: Images of the 319.1 alloy disc

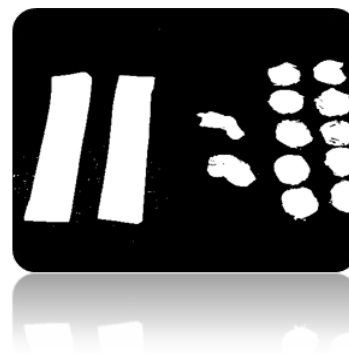


Figure 11b: A photograph of some shapes of the prepared Al 319.1 alloy

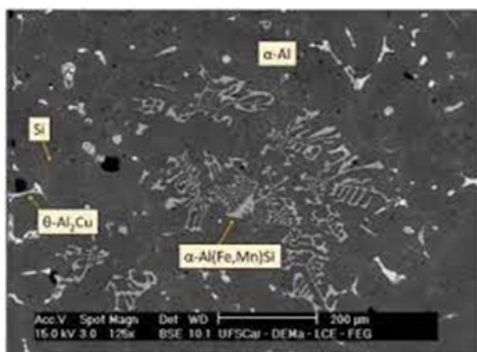


Figure 12: SEM of Al alloy 319.1 microstructure.

It can be seen in Fig. 12 that the alloying metals ≥ 0.5 Wt.% appeared in the scan image. Other minor elements are only detected in the XRD pattern given in Fig. 13.

Fig. 13 shows the XRD pattern of the prepared 319.1 alloy.

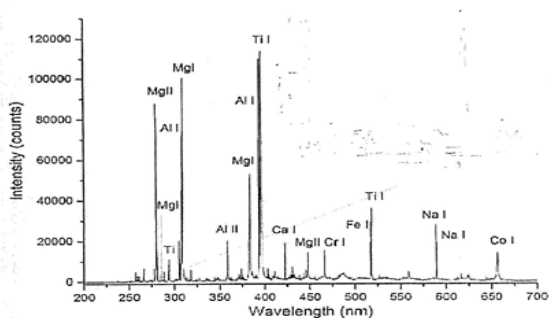


Figure 13: XRD pattern of the different compounds of the 319.1 alloy

Fig. 14 shows the XRD pattern of the different compounds of the 319.1 alloy after annealing at 850°C – 1100 °C for 10 h.

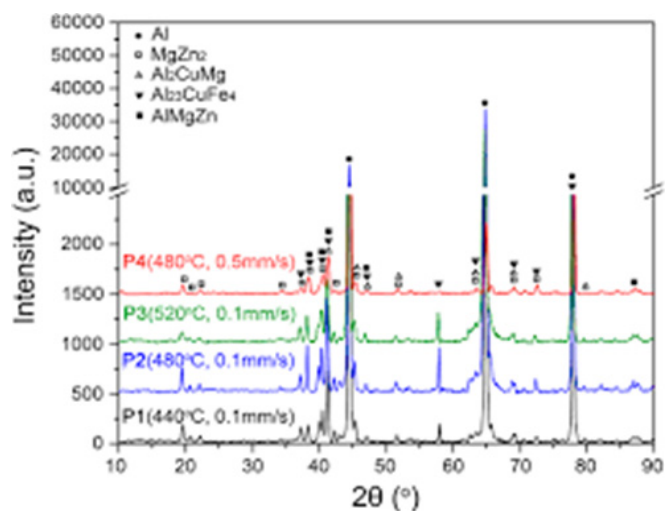


Figure 14: XRD pattern of the different compounds of the 319.1 alloy after annealing for 15 minutes at 850°C – 1100 °C for 10 h.

Conclusion

The output conclusion of this study can be summarized in the following. The metal turning scrap is a recurring source of metal values like copper, aluminium and nickel. There is no guarantee, during the metals machining, for assorting metal turnings from being mixed and wasted as a useless scrap. The weight percentage of the metals is a variable and differs from one workshop to another depending on the countryside and type of the industry. Direct melting of the scrap doesn't score the goal recovery of 319.1 alloys because of the broad variety of the melting point of the elements available. The suggested method involving magnetic separation of the ferromagnetic elements, hydrometallurgy selectively leaching of copper in alkaline ammonium hydroxide /ammonium bicarbonate solution containing 10 % H₂O₂, filtration of the reaction medium and leaching of the remaining unreacted metals with 3M HCl at room temperature becomes the suitable way to achieve preparation of the targeted Al 319.1 alloy. The precise and accurate composition of the 319.1 alloys is easily workable. Controlling of the portion of the metals solutions that are then mixed and reduced with 1 M ascorbic acid produces metal powders constituting the 319.1 alloy composition. Physical investigation of the recovered alloy revealed that the experimental route is successful.

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