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Tedd E. Lister, Peiming Wang, Andre
Anderko

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ELECTRORECYCLING OF CRITICAL AND VALUE METALS FROM MOBILE ELECTRONICS

*Tedd E. Lister¹, Peiming Wang² and Andre Anderko²

¹*Idaho National Laboratory*

P.O. Box 1625

Idaho Falls, ID 83404

(*Corresponding author: Tedd.lister@inl.gov)

²*OLI Systems, Inc.*

240 Cedar Knolls Road

Suite 301

Cedar Knolls, NJ 07927

ABSTRACT

Mobile electronic devices such as smart phones and tablets are a significant source of valuable metals that should be recycled. Value metals in phones are gold, palladium, silver, copper, cobalt and nickel. Devices now contain increasing amounts of rare earth elements (REE). Effective recycling schemes should include the recovery of these critical materials. Targeted recycling of items containing more of the less available critical materials could address their future criticality. This presentation will describe an electro-hydrometallurgy approach to efficient recycling of metals from scrap mobile electronics. The electrorecycling (ER) process generates oxidizing agents at an anode while reducing dissolved metals at the cathode. Development of the process involved generation of E vs pH diagrams, complemented with metals dissolution experiments used to assess the effectiveness of various solution chemistries. Although several schemes were envisioned, a two stage process has been the focus of work: 1) initial dissolution of Cu, Sn, Ag and magnet materials using Fe⁺³ generated in acidic sulfate and 2) final dissolution of Pd and Au using Cl₂ generated in an HCl solution. Results from laboratory work will be discussed.

KEYWORDS

Recycling, electrorecycling, electrowinning, electrodisolution, rare earth element, electronic scrap

INTRODUCTION

Mobile electronic devices such as smart phones and tablets are a significant source of valuable metals that should be recycled. Each year over a billion devices are sold world-wide and the average life is only a couple of years. To some extent, valuable metals are already recycled to specifically recover precious metals such as Au. Metals contained in phones, which can be called value metals, are gold, palladium, silver, copper, cobalt and nickel. These devices contain increasing amounts of what are called rare earth elements (REE). They are contained in displays, speakers and vibrators within the devices. Effective recycling schemes should include the recovery of these critical materials. By including more value materials in a recovery scheme, greater product diversification is achieved and less toxic material has to be disposed. Specific REEs are considered critical materials. The US Department of Energy (DOE) considers Nd, Dy, Eu, Tb, and Y as REE critical materials (DOE, 2012). Other REEs such as Ce, La, and Pr were deemed near critical. REEs are mined as a group where total REE concentrations in the ore are generally below 8% (Goonan, 2011). Many of the critical elements have low abundance in the ore. Thus, when specific elements become critical, significantly more ore must be processed to capture those dilute but valuable critical elements. Targeted recycling of items containing these critical materials could stem their future criticality. Many of these REEs are contained in mobile electronics, particularly Nd in magnets (Buchert et al., 2012). Recent reviews of REE reserves, suppliers, uses and potential recycling sources are available (Buchert et al., 2012; Schüller et al., 2011). REEs are used in increasing amounts in green energy technologies such as direct drive windmills and hybrid electric vehicle motors. Many devices that utilize large amounts of critical REEs have long service lives (windmills, electric motors) and thus are not currently available in significant amounts. Portable electronics, which have very short useful lifetimes, offer a modest content of critical REEs along with other value metals such as Au, Pd, Ag, Co, and Cu. Electrochemistry offers unique methods of enabling recovery of critical and value metals from mobile devices while minimizing chemical usage. This paper will review literature related to development of an electrochemistry based recycling process. Following this review, a discussion of process development will be presented as related to recovery of value and critical metals from mobile devices.

BACKGROUND

The following focused review covers areas of work pertaining to development of an electrorecycling process for mobile electronics. This review covers: 1) critical and value metals content in mobile electronics, 2) dissolution chemistry for electronics, 3) previous work in electrochemical recovery of metals and finally 4) dissolution and recovery of REEs. This review serves as a starting point in development of the process.

Mobile Electronics as a Source of Critical and Value Metals

Mobile electronics have changed dramatically in recent years. The size of circuitry has decreased as fewer discrete components are employed. Smart phones and tablets continue to grow in use (CCS Insight, 2013). These devices pack processors, displays, speakers, cameras, microphones, and other devices into a very compact space. Unlike personal computers (PC), component by component (display, speakers, hard drive, etc.) separation for targeted recycling of specific parts is less favorable due to small size and small amount of material per unit. As these devices are very recent, few recycling reports have specifically addressed recycling opportunities and schemes for them. In addition to value metals, they contain REEs in displays and speaker/vibrator/headphone magnets (Buchert et al., 2012). A USGS report assessed the metals content of mobile phones in 2006 (Sullivan, 2006). At the time a metals recycler estimated that 1 ton of cell phones contained 140 kg Cu, 3.14 kg Ag, 300 g Au, 130 g Pd and 3 g Pt. Precious metals comprise the greatest value of recoverable metals (~91%) (Hageluken, 2006; Cui et al., 2008). More complex devices available today are suspected to contain higher metal content per unit (Buchert et al., 2012).

The value metals within mobile electronics are contained primarily within circuitry and peripheral components such as displays and speakers. Cu traces connect circuit components and thus comprise a

large fraction of metal. Solder is used to form permanent electrical connections. Lead-free solder meets the Reduction of Hazardous Substances (ROHS) directive and contains Sn-Ag or Sn-Ag-Cu alloys. Surface contacts are coated with Au which may be deposited on interlayers such as Ni. Surface mount Ta capacitors have Ag and Pd electrodes. Magnets for speakers and vibrators are high strength Nd-Fe-B but contain Pr and possibly Gd and Dy in lesser amounts. Display backlighting in mobile devices use light emitting diode (LED) sources which contain small amounts of REE phosphors including Eu, Y and Tb (Buchert et al., 2012). Li-ion batteries used to power devices contain significant amounts of valuable Co. Thus, significant amount of critical, near critical, and value metals are present in these devices providing opportunities for economic and environmentally sound recovery methods.

Spent mobile electronics presents an enormous resource of critical and value metals. World sales for mobile phones (cell and smart phones) were approximately 1.7 billion in 2011 and 2012 (Gartner, 2013). The average device has a lifetime of 18 months in the United States (Environmental Protection Agency, 2004). Worldwide tablet sales for 2013 were estimated to be over 182 million, growing from 112 million in 2012 (Gartner, 2012). By the end of 2013 5.9 billion mobile devices are projected to be in use (CCS Insight, 2013). The worldwide combined sales of smart phones and tablets are expected to reach 2.1 billion in 2017. While some reuse of devices occurs, an increasing volume of mobile electronic devices will become available for recycling in the near future. Other devices such as e-readers, cameras, smart watches, and upcoming wearable electronics are also potential feedstocks for recycling efforts.

Dissolution of Electronic Materials

Metal dissolution is a principal step in hydrometallurgical processes. Significant work has been performed in the recovery of metals from electronics printed circuit boards (PCB) (Hagelken, 2006; Cui et al., 2008; Huang et al., 2009; Tuncuk, 2012). The methods used are analogous to those used in mining although the metals can be in higher concentration than in ore bodies (Huang et al., 2009). The dissolution of PCB metals has largely been performed using one of three acids: HCl, H₂SO₄, and HNO₃. The choice of acid is generally a balance of dissolution speed and compatibility with downstream processing of the leachate solution. Both HCl and H₂SO₄ may rely on dissolved O₂ as an oxidizing agent while HNO₃ is an oxidizer. The concentration of O₂ can be increased using pressure resulting in enhanced dissolution rates (Ruiz et al., 2007). Oxidizers, such as Cl₂, H₂O₂, and Fe⁺³ can be added to enhance dissolution rates (Tuncuk, 2012). Enhanced Cu dissolution using FeCl₃ has been demonstrated (Wang, et al. 2009). Cl₂ generated from mixing bleach and HCl is a powerful oxidizer (Herrerros et al., 1999). For dissolution of precious metals aqua regia (HCl-HNO₃) is effective, where NO₃⁻ or nascent Cl₂ acts as the oxidizer and Cl⁻ forms stable ion complexes (Cui et al., 2008). Thiourea ((NH₂)₂CS) with ferric oxidant is an alternative to cyanide that can be used to extract precious metals (Cui et al., 2008). Thiosulfate (S₂O₃⁻²) is another alternative leachate but is slow and has poor stoichiometric recovery (Cui et al., 2008). Complexing agents can stabilize solution species and reduce the required oxidizing strength. Cyanide has been used in mining activities for years but is considered environmental unfriendly (Cui et al. 2008). Molecular Cl, Br, and I are suited to dissolution of gold, acting as both the oxidizer and complexing agent. Using E vs pH diagrams, the order of stability for Au complexes increases in the order Cl, Br, I. Pd forms insoluble precipitates in neutral Br and I (Pourbaix, 1974). Thus Cl₂/HCl might be the best oxidizer/complexing agent for capturing both Pd and Au in a single step. Cl₂ is manufactured by the electrochemical chlor-alkali process or can be generated from mixing bleach with acid. Electrochemistry provides a controlled means for on-site generation. However, Ag forms insoluble compounds with halides complicating the use of single solution to dissolve all metals of interest.

Electrochemistry-Based Recovery of Metals

Electrochemistry-based metal recovery schemes have utilized two functions: 1) anodic generation of oxidizers to speed dissolution of metals from scrap or 2) cathodic electrowinning of leached metals for separation and recovery. In some cases, both functions have been combined to increase electron efficiency. This review will focus primarily on metals dissolution and/or recovery from electronic waste.

Electrowinning recovers dissolved metal ions by electroreducing to metal from leach solutions. Electrowinning for electronic materials involves reduction and deposition of metals at the cathode, often as a group where further processing is required to reach a final product. Electrowinning requires the reduction potential of the metal to be similar or more positive than the water reduction potential or primarily H_2 is produced. The anode reaction is typically O_2 evolution where durable anodes are employed. Ammoniacal solutions have been used to dissolve Cu as a copper amine complex from circuit boards followed by electrowinning (Oishi et al., 2007). Mecucci (2002) recovered Cu and Pb by electrowinning after dissolution of PCBs in HNO_3 where Sn precipitates as $H_2Sn_2O_3$ prior to plating. Cu, Sn, and Pb were recovered from PCB waste dissolved in sulfuric acid where deposits were initially rich in Cu followed by growing Pb and Sn content presumably due to depletion of Cu over time (Veit et al, 2006). Metals in industrial sludge were extracted into aqueous H_2SO_4 followed by electrowinning to recover Cu and Ni (Veglio et al., 2003).

Electrochemical processes have been developed to create a chemical environment to enable dissolution of metals. Protons can be generated at the anode through oxidation of water to O_2 to sustain the required pH to dissolve metals. While acids combined with dissolved O_2 have been shown to dissolve some metals effectively, oxidizers may be electrochemically generated to speed dissolution. These dissolution processes are analogous to electroplating methods utilizing a dissolving anode, except that the oxidizer/mediator acts as an electron shuttle in place of direct contact. This allows a complex mostly non-conductive matrix to be processed effectively. In situ generation of Cl_2 in HCl electrolytes has been reported for dissolving Cu in PCBs (Kim et al., 2008, 2010, 2011). This work used an anion exchange membrane separated cell with a graphite anode. Leaching was performed at room temperature due to the limited solubility of Cl_2 gas at elevated temperatures. They observed leaching of Pb, Sn, and Zn from PCB material approaching 100% with Cu being somewhat lower, attributed to $CuCl$ passivation (Kim et al., 2011). Ping et al. noted a significant increase in dissolution rate using electrogenerated Cl_2 versus dissolved O_2 from air as used for typical leaching (2009). Cl_2 may also be used to leach Au and Pd as described earlier.

It is possible, with appropriate chemistry, to perform dissolution and electrowinning functions using the same electrochemical cell, thus providing high electron efficiency. A Cl_2/Cl based system was modeled and examined for recovery of Au through combined dissolution electrowinning (Diaz et al., 1993). This work was expanded to include metals from electronic waste using electrogenerated Cl_2 to dissolve metals in a dissolution column while electrowinning metals as a group at the cathode (Brandon et al., 2001; Cheng et al., unknown). Analogous schemes have also been investigated for mining applications. Copper matte was electrorefined by generating Fe^{+3} at the anode to dissolve Cu and electrowin at the cathode (Jiricny et al., 2002, 2002). To achieve high Faradaic efficiency, chemistry and cell design must be carefully chosen. For example, poorly separated electrodes result in oxidizer discharge at the cathode leading to an effective short circuit in current. This phenomenon is known in electrowinning of Cu when Fe is present where efficiencies can drop to 60% (Beukes and Badenhorst, 2009). The reduction of Fe^{+3} can act as a competing cathode reaction where Fe^{+2} is formed without performing any desired chemical work. For an electrorecycling cell, this effect can be minimized by controlling flow or by employing ion-selective membranes to separate cathode and anode solutions.

Rare Earth Recycling Chemistry

Recycling methods for REEs are not as well developed compared to other value metals. Reviews have recently outlined potential REE recycling feedstocks, existing recycling technologies, and future needs (Binnemans et al., 2013; Tanaka et al., 2013; Anand et al., 2011). Nd-Fe-B magnet alloys dissolve rapidly in acid solutions and are typically coated with Ni or layers of Ni and Cu for protection. Selection of the dissolution chemistry depends on the anticipated recovery process. All REE form insoluble trihydroxides in basic mediums (Pourbaix, 1974). This can be used to precipitate REEs but requires a significant pH shift for acid-based leaching chemistries. The pH shift is also higher than commonly dissolved metals such as Fe and Ni. REEs are soluble in chloride and nitrate while having low solubility in sulfate except at low pH (Stevenson and Nervik, 1961). From moderate H_2SO_4 , a slight increase in pH to

slightly over 1 will precipitate the REEs as white double salt solids $((RE)_2(SO_4)_3 \cdot Na_2SO_4 \cdot xH_2O)$ using NaOH). This was used to capture REE from nickel-metal hydride (Ni-MH) batteries (Pietrelli et al., 2002; Bertuol et al., 2009). Pietrelli (2002) leached with 2 M H_2SO_4 resulting in over 90% REE release then increased the pH to 1.5 with NaOH to recover over 70% of REE content. REEs were recovered from magnet scrap (swarf) using sulfuric acid to dissolve followed by NaOH addition to form double salt precipitate (Lyman and Palmer, 1995). Phosphoric acid dissolution followed by pH increase has been used to precipitate REEs from Ni-MH battery HCl based leachate solutions (Lyman and Palmer, 1995). Fluoride and oxalate have both been used to precipitate REEs for separation (Tuncuk et al., 2012). Solvent extraction has also been used to recover dissolved REEs from Ni-MH batteries. Tzanetakis (2004) dissolved Ni-MH batteries in HCl and used 25% bis-(2-ethylhexyl) phosphoric acid (D2EHPA) in kerosene. Extraction at pH 2.5 removed nearly 100% REE, however, other metals were also extracted. Lamp phosphors show a wide variation in ease of dissolution (Binnemans et al., 2013). Specific recycling of REE from LEDs used for display backlighting was not found, possibly due to low amounts used. Nuclear fuels processing has also extensively studied solvent extraction for removing fission products (Stevenson and Nervik, 1961).

PROCESS DEVELOPMENT

INL Electro-Recycling System

The original concept drawing for the bench-scale INL (Idaho National Laboratory) electrorecycling system is shown in Figure 1. The system uses a single electrochemical cell with an oxidizer produced at the anode and dissolved metals reduced onto the cathode. The system uses a DC power supply to polarize the cell during operation. A redox probe was incorporated to track oxidizer strength during processing. The anode and cathode share a common reservoir but are separated by a diaphragm or anion exchange membrane to reduce cross-over. The exit from the anode is fed to a basket holding electronic scrap. The design shows a REE recovery system based on decreased solubility of REEs with a slight increase in pH. This original design went through changes to allow easier removal of the cathode by incorporating into a trough cell and removing cathode flow. The anode flow was controlled by pulling solution through the anode to the dissolver which drained back to the trough. One process issue is the need to add Cu salt or evolve H_2 at the cathode during startup until dissolved metal content increases. Future designs will utilize separate anode and cathode solutions to further decouple them. Solutions would: 1) dissolve metals as an anode solution followed by 2) metal electrowinning in the cathode compartment. Thus solutions would be swapped at intervals to increase efficiency. The cell will use an anion exchange membrane to limit crossover. Recovery of REEs will be through a membrane-based extraction being developed in another laboratory associated with the project.

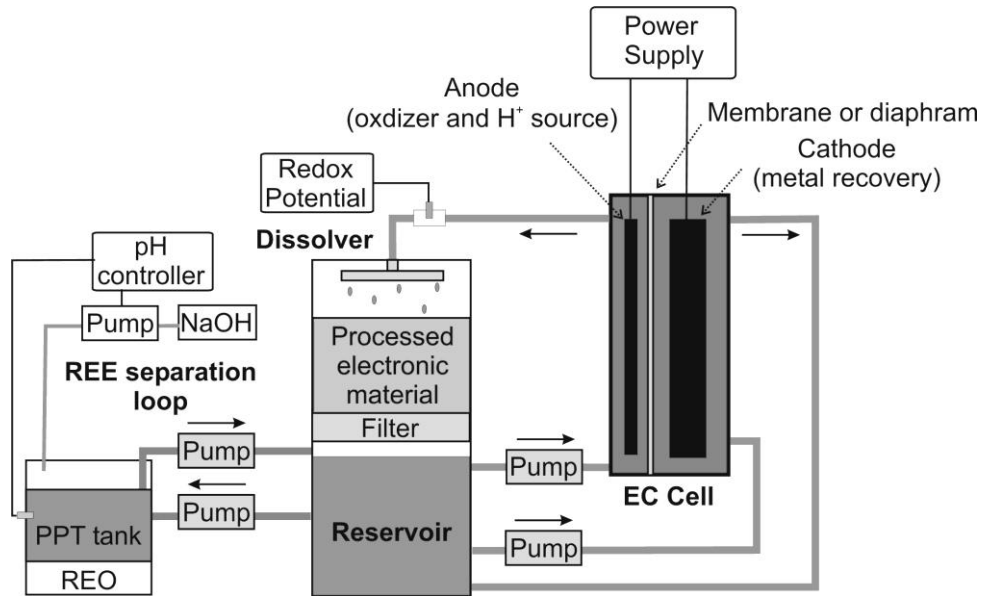


Figure 1 - Diagram of the electrorecycling system.

Preliminary Flow Sheet Discussion

Several flowsheets were envisioned in proposing to use electrorecycling for mobile electronics. The metals dissolved can be tailored by choice of oxidizer potential and E vs pH diagrams. A thermodynamic analysis and pure metal corrosion data was performed (Lister et al., 2014). It was found that Fe^{+3} in acidic sulfate solution at 60°C effectively dissolved Ag, Cu and Sn at rates exceeding 5 $\mu\text{m/hr}$. Both Au and Pd were unaffected under the same conditions. Using electrogenerated Cl_2 gas fed to an HCl dissolution solution, Au and Pd can be effectively dissolved. Based on these results, preliminary flow sheets were developed with an attempt to reduce the number and complexity of pre-processing steps, where the material is crushed to small particles.

Using data accumulated from dissolution of pure metals three potential flow sheets were considered for future work. The first flow sheet shown in Figure 2 uses a single electrorecycling stage to dissolve Fe, Sn, Cu and Ag while leaving Au and Pd in the matrix. The electronic scrap would be treated to a more robust oxidizer solution to dissolve Au and Pd. REEs would be removed from the electrorecycling solution by extraction or precipitation. A variation on this method would employ a second electrorecycling stage to remove precious metals as shown in Figure 3. This would involve anode generation of Cl_2 and cathode reduction of dissolved metals. While this would be a viable method it would involve additional capital expense per volume of material processed as either a second type of cell would be needed or a single cell would perform both reactions and thus be drawn away from the primary dissolution/recovery step. Given the lower amounts of precious metals present and high value, it may be economical to purchase an oxidizer or use a smaller on-site chlor-alkali system to produce oxidizer. With either of these methods metals would be partitioned into three types: REEs, non-noble metals, and noble metals. A third approach, shown in Figure 4, utilizes a single strong oxidizer. Such a system was conceived of previously for electronic waste dissolution and recovery using Cl_2 (Brandon et al., 2001; Cheng et al., unknown). The important issue, not discussed in the previous work, is the formation of insoluble AgCl which would need to be stripped from the remaining waste afterwards. Thus Cl_2/HCl would not be a true single solution approach. This type of cell would require controls to deal with Cl_2 as both a possible off-gas or and require more robust materials for construction.

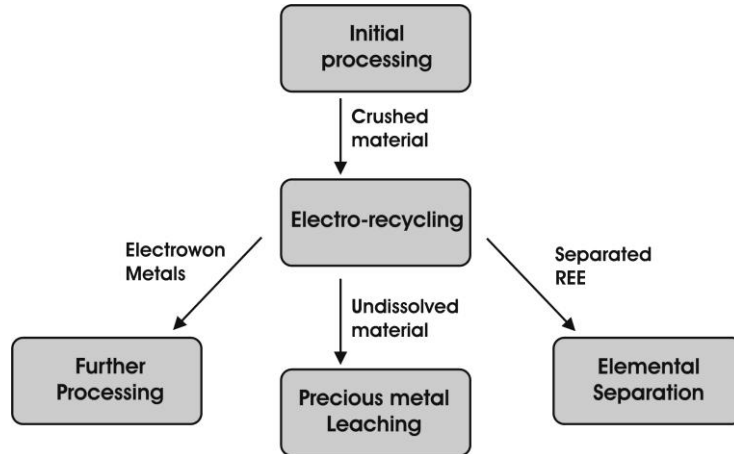


Figure 2 - Proposed flow sheet for a single mildly oxidizing electrorecycling stage followed by chemical dissolution of precious metals.

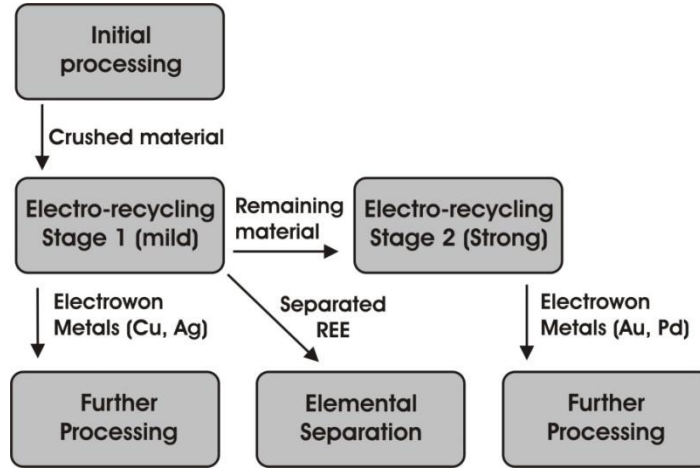


Figure 3 - Proposed flow sheet for a two stage recycling scheme: 1) mild oxidizer dissolution (Fe^{+3}) stage followed by 2) strong oxidizer (Cl_2) dissolution stage.

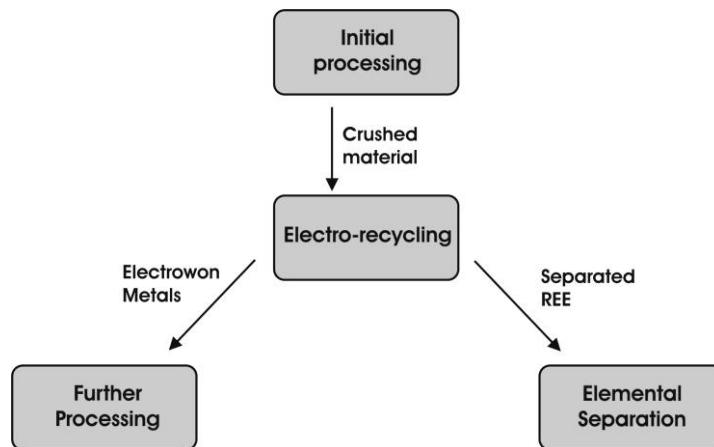


Figure 4 - Proposed flow sheet for a single electrorecycling stage utilizing a strong oxidizer (Cl_2).

CONCLUSIONS

Mobile electronics offer great potential for recovery of value and critical metals provided effective chemistry is developed. Electrorecycling could minimize chemical inputs to dissolve metals while simultaneously recovering them on a cathode or extraction method. A combination of thermodynamic and experimental data was used to support development. Dissolution in oxidizing solutions provided data for rates of dissolution for various metals of interest for recycling. A discussion of recycling schemes was presented using both mild and aggressive electrogenerated oxidizers. To recover value metals, it appears that a minimum of two stages are required.

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